Operator's Manual

Du Pont Instruments

983 Dynamic Mechanical Analyzer

PN983006.001 Rev. B
Issued November 1986

(c) 1986 by E.I. du Pont de Nemours & Co. (Inc.)
Concord Plaza - Quillen Building Wilmington, DE 19898

NOTICE

The material contained in this manual is believed adequate for the intended use of this instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their validity and suitability must be obtained from Du Pont. Otherwise, Du Pont does not guarantee results and assumes no obligation or liability. This publication is not a license to operate under nor a recommendation to infringe upon any process patents.

Notes, Cautions, and Warnings are used in this manual to emphasize important and critical instructions under the guidelines described below:

WARNING

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

CAUTION

A CAUTION emphasizes a procedure that may damage equipment or lose data, if not followed correctly.

NOTE

A NOTE highlights important information about equipment or procedures.

Thermal Analysis Hotlines

Technical Assistance	302-772-5134
To Order Instruments and Supplies	302-772-5500
Service Inquiries	302-772-5576
Sales Inquiries	302-772-5488

Safety Notes

Electrical Safety

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

WARNING

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

Handling Cryogenic Materials

When you use the 983 DMA system to perform subambient experiments, the 983 will use the cryogenic (low temperature) agent, liquid nitrogen, for cooling. Because of its low temperature (-195°C), liquid nitrogen will burn the skin. Personnel working with liquid nitrogen should take the following precautions:

WARNING

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

1. Wear goggles or a face shield, gloves large enough to be removed easily, and rubber aprons. For extra protection, wear high-topped shoes with pant legs outside the tops.

2. Transfer the liquid slowly to prevent thermal shock. Use containers that have satisfactory low-temperature properties. Make sure that closed containers have vents to relieve pressure.

3. The purity of a container of liquid nitrogen alters as the nitrogen evaporates. If much of the liquid in the container has evaporated, check the remaining liquid because it might have a dangerous oxygen content.

If a person is burned by liquid nitrogen:

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

2. If the skin is blistered or if there is a chance of eye infection, get the person to a doctor immediately.

Using This Manual

This manual contains all the information you will need to operate the 983 Dynamic Mechanical Analyzer.

CHAPTER 1	**introduces** the system with a description of the system and its specifications.
CHAPTER 2	contains the **installation instructions** and information about **getting started.**
CHAPTER 3	describes **routine operation** of the 983 system including **subambient operations.**
CHAPTER 4	provides **technical information** and additional operating instructions for the system.
CHAPTER 5	contains basic **maintenance** procedures and test codes.
APPENDIX	contains helpful reference material and additional information regarding **error codes,** and options information.
GLOSSARY	defines important **terms** used throughout this manual.

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CHAPTER 1

Introducing the 983 DMA

Introducing the 983 DMA

Overview

The Du Pont 983 Dynamic Mechanical Analyzer (DMA) is a highly sophisticated mechanical analysis module that can be used with the Du Pont 9900 Thermal Analyzer running at least version 6.0 software. The 983 DMA is an instrument that offers a rapid and sensitive means to simultaneously obtain an elastic modulus (stiffness) and a mechanical damping (toughness) for materials. The 983 DMA has three major parts: the drive assembly where the sample is clamped and enclosed by the oven, the oven where the temperature is controlled, and the DMA base where the system electronics are housed.

The 983 DMA module measures changes in the viscoelastic properties of materials resulting from changes in temperature, frequency, and time. There are four modes of operation: resonant frequency, fixed frequency, stress relaxation, and creep. Each of these modes measures different aspects of the viscoelastic properties.

Principles of Operation

The sample is clamped between two parallel arms and is deformed under a constant stress, oscillating stress or a constant strain, depending on the experiment mode. The behavior of the sample under this deformation is monitored by a linear variable displacement transducer (LVDT). The following sections describe the four modes of operation.

Resonant Frequency Mode

When operating the 983 DMA in the resonant mode, the sample and arms form a compound resonance system. The sample is displaced and set into oscillation. Normally, a system so displaced would oscillate at the system's resonant frequency, with constantly decreasing amplitude due to the loss of energy (damping) within the sample. The amplitude signal from the LVDT is used to control the output signal of the electromechanical driver. The driver supplies additional energy to the driven arm forcing the coupled system to oscillate at a constant amplitude.

The frequency of oscillation is directly related to the stiffness or storage modulus of the sample under investigation, while the energy needed to maintain constant oscillation amplitude is a measure of the damping within the sample.

Fixed Frequency Mode

The fixed frequency mode is similar to the resonant frequency, except that the oscillation frequency is fixed. The sample is forced to undergo oscillatory motion using a sinusoidal driver signal. The sample displacement is monitored by the LVDT and the lag between the driver signal and the LVDT is the phase angle. The phase angle and drive signal are used to calculate the storage, loss modulus, and tan delta of the sample.

Stress Relaxation Mode

In the stress relaxation mode, the sample is flexed by displacing the arm position a specified amount. The amount of power required to maintain the selected position is then monitored as a function of time. Additionally, sample recovery is measured when the sample is released to an unstressed state.

Creep Mode

In creep, the fourth mode, the sample is displaced but the force is held constant. The position is then monitored as a function of time. Once again, the sample is released to an unstressed state and sample recovery is monitored.

Quantitative Calculations

The 983 DMA module provides real time quantitative calculations of the viscoelastic parameters. These values are also passed to the 9900 system for display and storage. The standard calculations for fixed and resonance modes are:

- E' (flexure storage modulus)
- Log E'
- E" (flexure loss modulus)
- Log E"
- Tan delta (loss factor, E"/E')
- Log tan delta
- G' (shear storage modulus)
- Log G'
- G" (shear loss modulus)
- Log G"
- Frequency
- Drive Signal
- Phase Angle

Additional data analysis programs are available on the 9900 control system to calculate compliance values, create master curves, and calculate kinetics parameters.

The 983 DMA and Accessories

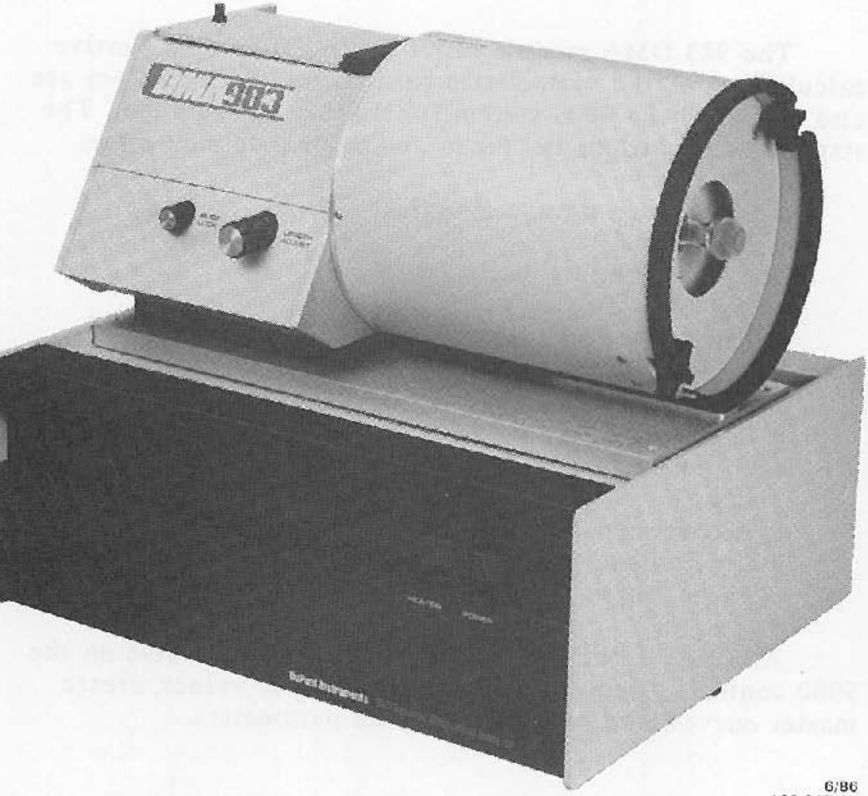

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Figure 1.1
983 Dynamic Mechanical Analyzer

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Figure 1.2
The 983 Dynamic Mechanical Analyzer with Local Keyboard

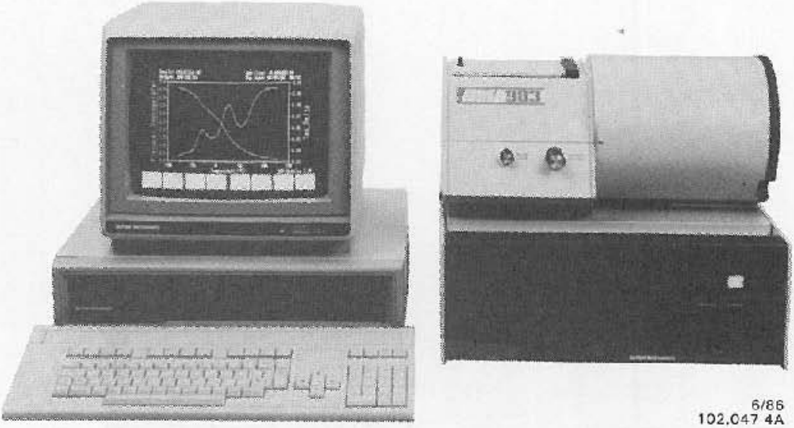

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Figure 1.3
The 983 Dynamic Mechanical Analyzer with 9900 Thermal Analyzer

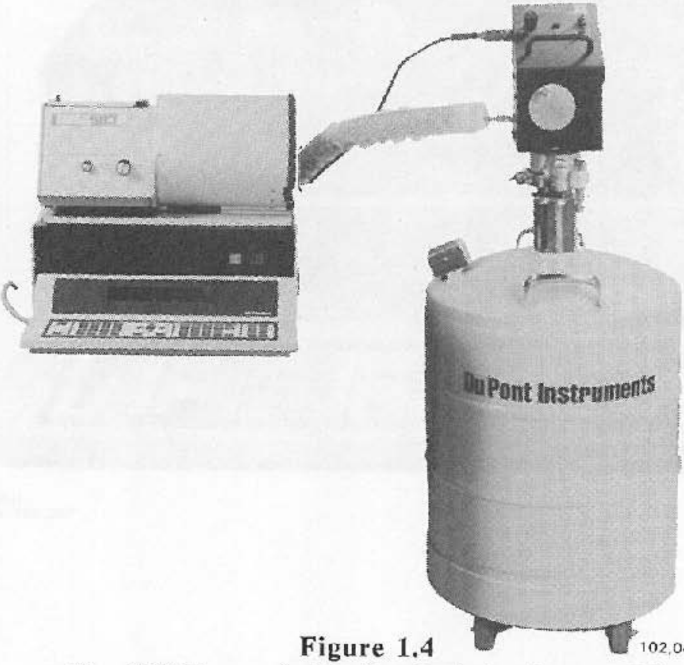

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Figure 1.4
The 983 Dynamic Mechanical Analyzer with the Liquid Nitrogen Cooling Accessory

System Components and Accessories

The 983 Dynamic Mechanical Analyzer is composed of three parts: the drive assembly, the oven, and the DMA base. See Figure 1.5. The drive assembly contains the electromagnetic driver, the flexure pivots, and the linear variable displacement transducer (LVDT). See Figure 1.6. The electromagnetic driver displaces and drives the sample-arm system; the LVDT measures the arm position.

The oven contains a radiant heater and a system for the distribution of coolant. A thermocouple located close to the sample senses the temperature and transmits this information to the control electronics located in the DMA base. Inside the oven, the sample is clamped between the free ends of the arms.

The DMA base contains the electronics and software needed to control the system, perform experiments, and store experimental results. If the power is interrupted, parameters vital to system operations are saved in battery backed-up RAM. The DMA base also contains additional RAM memory for local data storage and a GPIB interface for communications with the 9900. The keyboard/display unit connects to the back panel of the 983 DMA base allowing local control of experiments on the 983 DMA module.

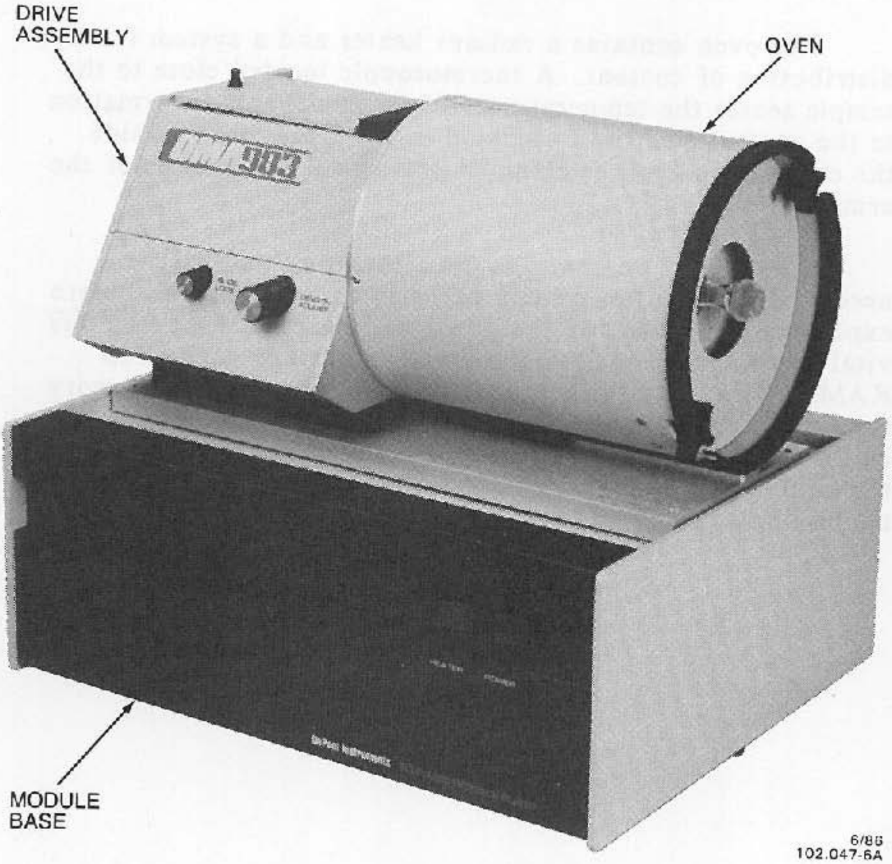

Figure 1.5
983 Dynamic Mechanical Analyzer

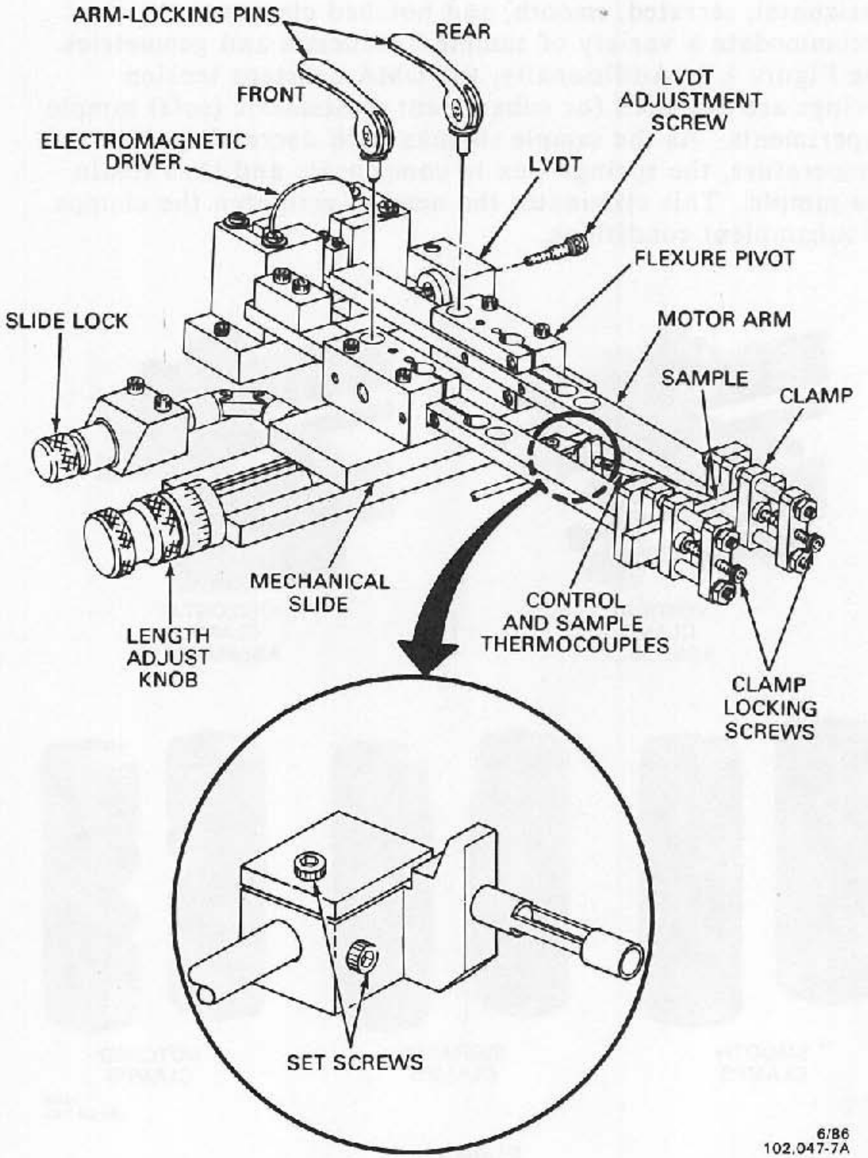

Figure 1.6
DMA Internal Components

983 DMA Clamping System

The clamping system for the 983 DMA includes vertical, horizontal, serrated, smooth, and notched clamps that accommodate a variety of sample hardnesses and geometries. See Figure 1.7. Additionally, the DMA constant tension springs are designed for subambient elastomeric (soft) sample experiments. As the sample shrinks with decreasing temperature, the springs flex to compensate and thus retain the sample. This eliminates the need to retighten the clamps at subambient conditions.

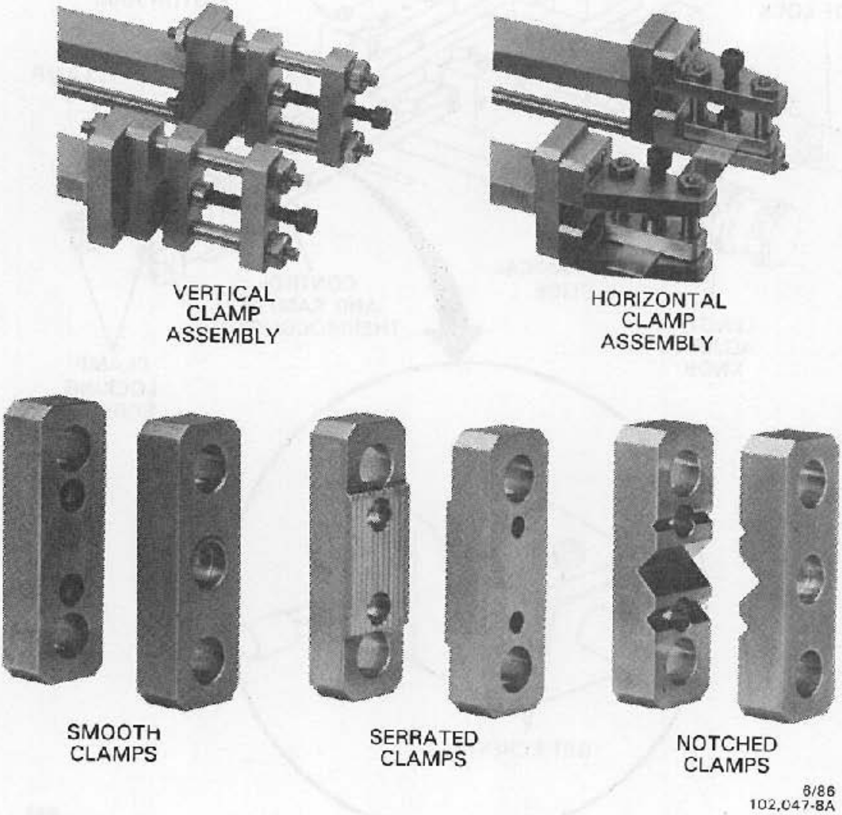

Figure 1.7
Clamp Configurations

Table 1.1
Measurement Ranges

Modulus Range	1 MPa to 200 GPa
Modulus Precision (5-30 Hz Resonant)	5%
Tan delta:	
Resonant Frequency	0.002 to 1.0
Fixed Frequency	0.002 to 10.0
Amplitude Range (peak to peak)	0.1 to 2.0 mm
Resonant Frequency	2 to 85 Hz
Fixed Frequency Range	0.001 to 10.0 Hz
Phase Angle Range	0.0 to 2.8 rad
Phase Angle Precision	0.001 rad (0.06 deg)
Stress Relaxation Drift (at 300°C for 1 hour)	< 5%
Creep Drift (at 300°C for 1 hour)	< 5%
Temperature Range	ambient to 500°C
with LNCA	-150 to 500°C

Table 1.2
Sampling System

Length Range	6 to 65 mm vert. 6 to 57 mm horiz.
Max. Thickness	12 mm vert. 12 mm horiz.
Max. Width	15 mm vert. 5 mm horiz.

Table 1.3
Temperature Control

Program Heating Rate	0.01 to 50°C/min.
Isothermal Stability at 100°C for 1 hr. subambient	 ±0.1°C ±1.0°C
Temperature Precision	±0.1°C
Programmed Cooling Rate with LNCA	1 to 5°C/min. to -150°C
Cool Down Time (Ambient to -120°C with the LNCA)	15 min.
Purge Gas	N_2 or air

Table 1.4
Power Requirements

983 DMA w/o LNCA	115 V ac 50/60 Hz 900 watts
with LNCA	1250 watts

CHAPTER 2

Installation and Keyboard Operation

Installation and Keyboard Operation

Installation

Installing the 983 DMA is a simple task. To avoid mistakes, read this entire section before you begin. When you receive your 983 DMA, look over the instrument and shipping container carefully for signs of shipping damage, checking the parts received against the enclosed shipping list. If the instrument is damaged, notify the carrier and Du Pont Instruments immediately. If the instrument is intact but parts are missing, contact Du Pont Instruments. A list of Du Pont offices can be found on the last page of the appendix in this manual.

Choosing a Suitable Location

> **NOTE**
>
> **Be sure to place the DMA on a level, vibration-free surface. Unsteady surfaces can interfere with DMA measurements and cause signals to be unstable.**

The 983 should be placed on a level, vibration-free surface. The module operates best in a clean environment. Ample work space is necessary. Choose a sturdy bench or table near power outlets which have the proper voltage, frequency, and current ratings. The 983 DMA is designed to operate at 115 V ac, 50 or 60 Hz, at 15 amps. A step up/down power line transformer may be required if the unit is operated at a higher or lower line voltage. Also, make sure the power and communication cable paths are protected; do not create hazards by laying cables across pathways or access ways. Avoid dusty, drafty, and poorly ventilated areas as well as locations with direct sunlight exposure. Locate the 983 DMA near the 9900 computer thermal analyzer system.

The 983 DMA is shipped ready for operation, except for hookup to the 9900 Thermal Analyzer, keyboard, and LNCA.

> **NOTE**
>
> **Connect all cables before connecting the power cord to the outlets.**

Connecting the GPIB Cable to the 9900

To connect the GPIB cable from the back of the module to the 9900, locate the GPIB connector. Find the GPIB cable. It is the only one which fits into the connector marked GPIB. See Figure 2.1.

> **NOTE**
>
> **Be sure to tighten the GPIB connector before using it.**

Select an address between 1 and 9 inclusively. If the address is changed after the 983 is powered on, the 983 reset button must be pressed to enter the new address.

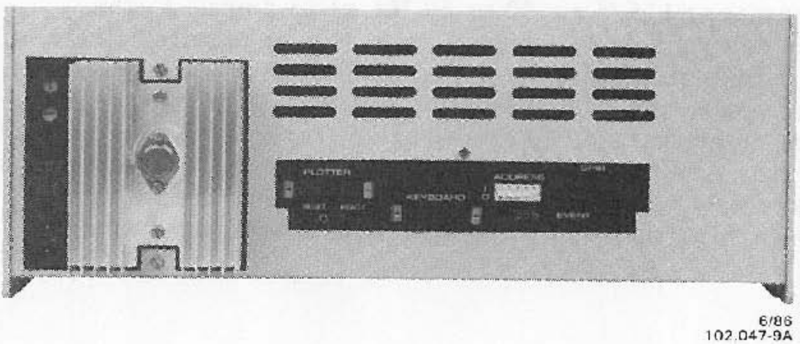

Figure 2.1
Rear View of the 983

Table 2.1
Possible Switch Patterns

Address	Switch Pattern
1	00001
2	00010
3	00011
4	00100
5	00101
6	00110
7	00111
8	01000
9	01001

Connecting the 983 to the Local Keyboard

First, be sure that the power is OFF.

CAUTION
Never connect or disconnect the keyboard cable while power is applied to the 983.

The keyboard plugs directly into the keyboard connector found on the back of the module. Each connector is simple to find and is keyed so each individual cable fits into its own connector.

Connecting the 983 to the Power Cable

NOTE

Connect all cables <u>before</u> connecting the power cord to the outlets.

To connect the 983 DMA to the power cable, plug the power cord into the 983 and with the module **POWER** switch in the OFF (0) position; plug the power cord into an outlet.

CAUTION

Before attaching the 983 power cord to the wall outlet, make sure the instrument is compatible with line voltage. Check the label on the back of the unit to verify the voltage.

When you are ready to make a run, you will also need to turn on the 983 **HEATER** switch.

NOTE

The HEATER switch will not light immediately. It will only light after a method is started.

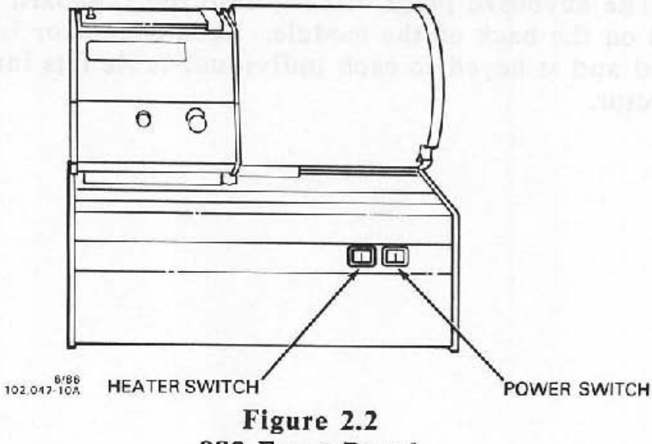

Figure 2.2
983 Front Panel

Connecting the 983 to the LNCA

If you wish to hook up the Liquid Nitrogen Cooling Accessory at this time, refer to the instructions in the manual that accompany the accessory.

When connecting the 983 DMA cable to the LNCA, the 983 power should be off. Use the cable provided to plug the two together. Refer to the LNCA manual for more information.

You are now ready to power-up the 983 DMA. The power-up procedure is described in the next section. Experimental set-up procedures are described in Chapter 3, Running a Sample.

Powering-up

Starting the Module

After the installation of the 983 DMA is completed, you are ready to start up the module.

> **NOTE**
>
> **Allow the 983 to warm up at least 30 minutes before performing an experiment.**

1. Check all connections between the local keyboard, if present, the 983, and the 9900. Make sure that each component is plugged into the correct connector.

2. Turn the **POWER** switch to the ON position. See Figure 2.2. The READY light (Figure 2.1) should come on after 2 to 6 seconds and glow steadily. Watch the display, if attached, during the confidence test for error indications. If everything is functioning properly or no <u>fatal</u> errors have been detected, the Du Pont Title screen appears on the module keyboard. See Figure 2.3.

```
128K          983 DMA Module            GPIB-1
Copyright 1986 E. I. DuPont Company Inc.
```

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Figure 2.3
Module Opening Screen

The screen presents the current system configuration: 128K of available data storage memory and a GPIB address set to 1. Your system's message may vary as it reflects options installed in your 983 module.

The mode selection screen appears three seconds after the title screen. See Figure 2.4. Turn on the **HEATER** switch (Figure 2.2) if you want to perform an experiment. See page 2-4 for NOTE.

```
Select Run Mode:                    Temp °C
>Resonant Frequency                   19.81
```

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Figure 2.4
Mode Selection Screen

Understanding the Confidence Test

Every time the 983 DMA is powered up or reset, the instrument automatically performs an internal confidence test.

One board contains all the major logic functions. Additionally, there are two optional subassemblies available for the 983:

1. The RAM memory expansion option, which increases the storage area for experimental data from the standard 128 kilobytes up to a total of 256 kilobytes.

2. The local keyboard and display assembly.

If any of these options is installed, the confidence test automatically tests it.

The confidence test checks all of the essential logic circuitry in the 983 in the following preprogrammed order:

- CPU control logic;
- Saved Parameter storage area;
- General data storage area;
- Program storage area;
- Major instrument control I/O chip functions;
- Run data storage area;
- RAM expansion board (if installed);
- Local Keyboard/Display (if connected);
- GPIB interface; and,
- Saved parameters checksum.

Become familiar with the testing order so you can determine what failed, what passed, and what has not yet been tested should an error occur during the confidence test.

The confidence test takes between 2 - 6 seconds to complete, depending on the number of installed options. If the test is completed without a fatal error, the READY light on the rear panel glows steadily. If the READY light fails to come on, a fatal hardware error has been detected. If the 983 detects an error during the confidence test, the instrument finds and identifies the problem on the local keyboard screen. The screen lists the hardware error and identifies whether the error is fatal or not. As the confidence test is in progress, a series of numbers flash on the far right of the screen. Each of these numbers designates a particular area within the instrument. If an error occurs, check the list of error codes and descriptions in Chapter 5. If there is a fatal error, contact your Du Pont Service Representative.

Shutdown

The 983 Dynamic Mechanical Analyzer electronics perform more reliably if power fluctuations are minimized. Therefore, turning the system on and off frequently is discouraged. If it is necessary to shut down, turn the **HEATER** and **POWER** switches off on the front panel. The READY light and display will go out when the power is off.

Working from the 9900

When working from the 9900, your thermal analysis system is a network with the computer at the center. Always power up the network in the following order: module(s), interface(s) and other devices on the GPIB, printer(s) and plotter(s), and finally the computer. The computer should be started last to minimize circuitry damage caused by power surges.

When you turn on the power to the computer, the system runs a confidence test. If all is well, a start-up program loads automatically from the disk.

The TA Operating System Loader screen offers two options:

- Press **F1** to load the DuPont Thermal Analysis Operating System and the programs needed for instrument control and data analysis.

- Press **F2** to select the MS-DOS Operating System. The E> prompt indicates that you are accessing the hard disk. An A> prompt indicates the current working drive is the left-most floppy diskette drive. From this point, you can load and run any program written under MS-DOS by using standard MS-DOS commands. Refer to the MS-DOS Operating System manual for more information.

NOTE

If you are currently in MS-DOS or if you made a mistake in the power-up sequence, press the CTRL (control), ALT, and DEL keys simultaneously to reset the system. This sequence is equivalent to, and safer than, turning the power off and on. These keys may be used to reset the system at any time except during the loading of the Du Pont TA OS.

CAUTION

Never reset the system (CTRL + ALT + DEL) or remove power when any disk drive red activity light is on. Resetting the system during disk activity may damage the data and programs stored on the active diskette. Wait for all disk activity to stop before resetting the system.

When selecting **F1** from the TA Operating System Loader screen, the System Configuration screen appears. See Figure 2.5. The display includes the date, time, and a table of the modules currently in the network. Across the top of the screen is a line showing the status of the Thermal Analysis module(s): type, temperature, gas status, event status, etc. A green box highlights the selected module number on multimodule set-ups.

The table headed "Address. . .Status. . ." represents the devices that the computer identified on the GPIB at start-up. The address is the GPIB address set on the switches on the 983. Refer to Table 2.1, page 2-3. Status is online or offline. The unit must be powered up and ready at start-up to appear on the table. For further information refer to the 9900 manual.

Master Screens

When using the 9900 with the 983 DMA, the system is organized around three master screens:

1. The **System Configuration** screen appears when the TA Operating system first loads (function **F1** from the TA-OS Loader screen). Use this screen to add or delete devices from the GPIB network, turn the status line on or off, etc.

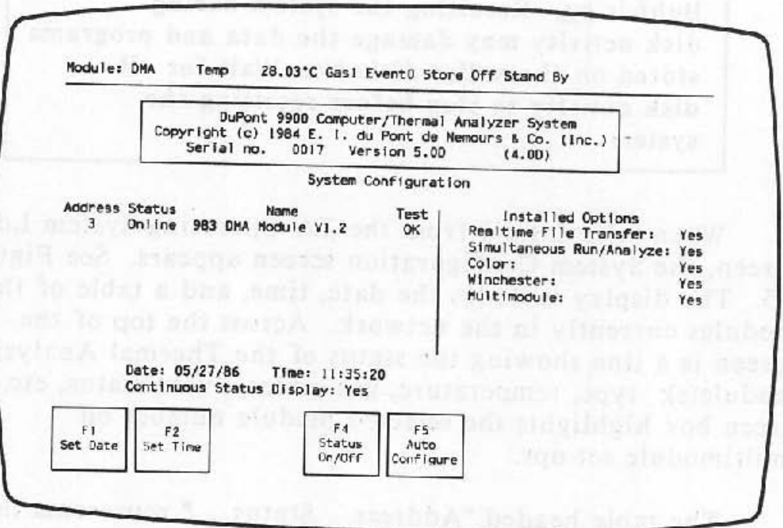

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Figure 2.5
System Configuration Screen

2. The **Instrument Control** screen appears when you press the **INSTR CONTROL** key, **F12**. This is the entry into the array of functions and screens that selects modules, module parameters, methods, etc.

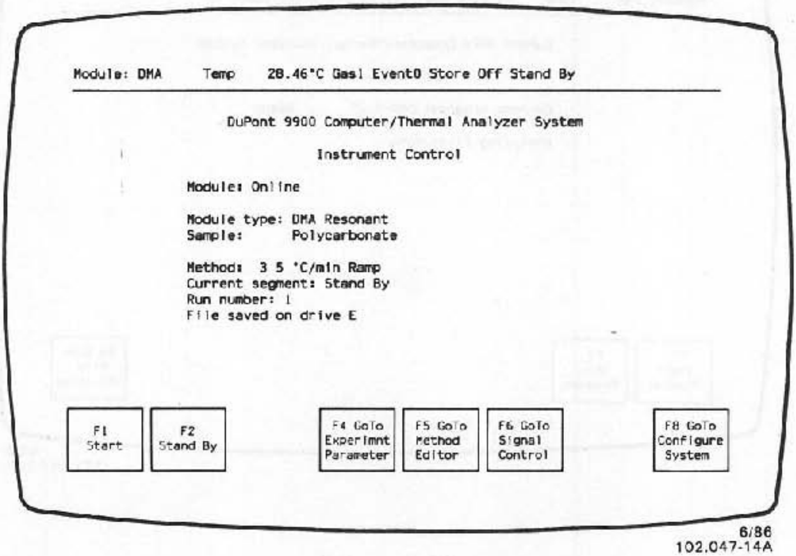

Figure 2.6
Instrument Control Screen

3. The **Data Analysis** screen appears when you press the **DATA ANALYSIS** key, **F11**. This screen allows you to set up, load, and run Du Pont TA Data Analysis and Utility Programs.

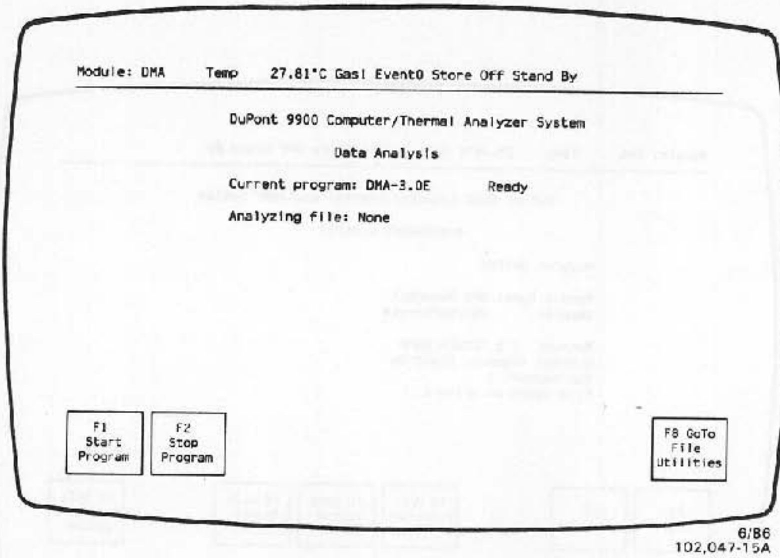

Figure 2.7
Data Analysis Screen

Function Keys

Every screen has a row of blocks (called Variable Function blocks) across the bottom labeled **F1, F2,** etc. There can be up to eight of these blocks, but they are all rarely displayed at once. The software is designed to show you only the choices that are logical progressions from the current system state. When you select a function key, a new screen and a new set of functions appear. To return to the previous screen, press **ESC**.

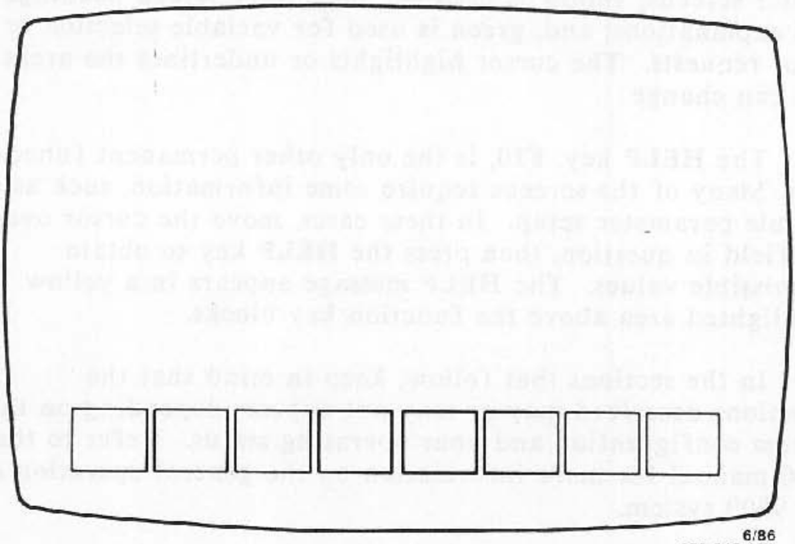

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Figure 2.8
Variable Function Block

The function keys **F11** (**DATA ANALYSIS**) and **F12** (**INSTR CONTROL**) return you to their respective Master screens at any time: **F11 and F12** are permanent, not variable, function keys.

Function key blocks appear in two colors on color monitors. Instrument Control screens have green function blocks, and Data Analysis screens have blue function blocks.

Color also provides a clue to the type of information presented on the screen: light blue (cyan) indicates titles or Master screens; yellow is used for subsidiary screen headings and explanations; and, green is used for variable selection or input requests. The cursor highlights or underlines the areas you can change.

The **HELP** key, **F10**, is the only other permanent function key. Many of the screens require some information, such as a module parameter setup. In these cases, move the cursor over the field in question, then press the **HELP** key to obtain permissible values. The **HELP** message appears in a yellow highlighted area above the function key blocks.

In the sections that follow, keep in mind that the functions described may or may not appear, depending on the system configuration and your operating status. Refer to the 9900 manual for more information on the general operation of the 9900 system.

```
Select Run Mode:                 Temp °C
>Resonant Frequency                19.81
```

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Figure 2.9
Mode Selection Screen

When working the module from the local keyboard, the display (often called the "screen") is the green lighted area of the keyboard unit designed to facilitate operator-983 communications. The screen contains two rows of 40 characters each. When additional information must be shown, the previous lines are cleared and new lines appear.

During normal operation, the display is clearly segmented into two areas. The 32-character section on the left is the interactive area. The right-most eight characters on both lines are reserved for online status messages. **HELP** and **ERROR** messages are exceptions to the divided screen; they utilize the entire display area. Status codes are described in Chapter 4.

When the 983 unit needs information, it displays a request on the screen followed by a default answer and prompt. To select the default shown, press the **ENTER** key. To select another response, use the numeric, alpha, **INC**, **DEC**, **YES**, or **NO** keys. The cursor prompt provides a clue to the type of response required: a flashing underline (_) indicates a numeric or alphanumeric entry; a greater-than symbol(>) indicates that multiple choices exist which you may review or select by scrolling. Additional assistance is available for every request and display. To obtain help, press and hold the **HELP** key.

Description of the Local Keyboard

The keyboard is divided into four groups according to functions:

1. Keyboard controls,

2. Entry keys,

3. Parameter keys, and

4. Operate keys.

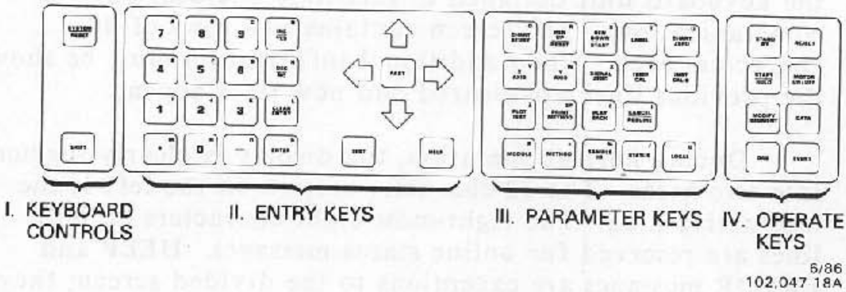

Figure 2.10
The Keyboard

The local keyboard/display unit permits communication between you and the 983. The keyboard may be used to:

1. Enter alphanumeric information.

2. Control the cursor position on the display.

3. Obtain assistance.

4. Perform system function tests.

5. Set up and run thermal programs.

6. Obtain a real-time status display.

7. Control experiment parameters and events.

8. Reset the system.

Keyboard Controls

The two keyboard controls are located on the far left side of your keyboard. Their functions are described below.

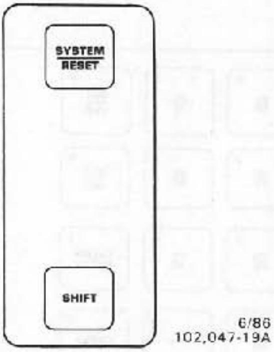

Figure 2.11
Keyboard Controls

The **SYSTEM RESET** key pressed with the **SHIFT** key interrupts the current activity and returns the system to the initial power-on state. The **RESET** button on the back of the 983 performs the same function.

NOTE

A system reset deletes all stored run data.

The **SHIFT** key is used to enter the letters, spaces, or symbols printed in red in the upper right corner of the entry and parameter keys. The **SHIFT** key must be pressed and held with the desired character key.

Entry Keys

Letters A-P are arranged alphabetically on the left portion of the entry key section. Q-Z share the parameter keys. Use the **SHIFT + UP/DOWN ARROW** keys to change from upper to lowercase and vice versa for the current entry. Uppercase is initiated automatically at start-up, after a reset, and at the beginning of each new entry.

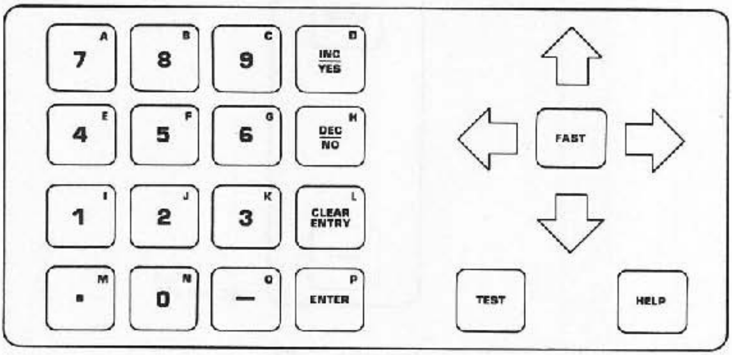

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Figure 2.12
Entry Keys

NOTE

The SHIFT key must be pressed and held to enter letters, spaces, and symbols.

Spaces and symbols are also included for text entries:

- Left parenthesis
- Right parenthesis
- Number of pound sign
- Plus sign
- Percent sign
- Slash
- Degree symbol

The **INC** and **DEC** keys have two functions. They are primarily used to increase or decrease numeric values. Additionally, they are used to scroll through multiplechoices. When more than one choice is provided, the system displays a greater-than symbol(>) as a prompt. In these cases, the **INC** key moves the cursor to the next selection; the **DEC** key moves to the previous choice. The responses are circular, i.e., you may move quickly to the end of the list from the beginning by pressing the **DEC** key; move to the beginning of the list from the end by pressing **INC** key. Press **ENTER** once you have scrolled to the desired answer.

NOTE

The SHIFT key is not required to use INC or DEC; the system automatically selects the correct function for the key.

The **YES** or **NO** key is used to answer questions requiring this response. The **YES** and **NO** keys answer and enter the question response in one keystroke; you do not have to press **ENTER** after the **YES** or **NO** key.

CLEAR ENTRY erases the current entry and sends the cursor to the left-most position. If you press **CLEAR ENTRY** twice, the current entry is erased and replaced with the original value.

The **ENTER** key inputs numeric text or preprogrammed responses to the module. After you press **ENTER**, the cursor moves to the next field.

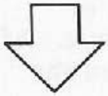
Displayed numeric parameters can be rapidly increased or decreased by using the **INC** or **DEC** keys while the FAST key is pressed and held. **FAST** has no effect on the scrolling function of the **INC** or **DEC** keys. The **FAST** key is also used with the **LEFT** and **RIGHT ARROW** keys to move the cursor rapidly through an alphanumeric field, right or left 10 characters at a time.

Cursor Control

The arrow keys move the cursor and are used for editing. They may be used alone to move the cursor up, down, right, and left through the text. Used in conjunction with the **SHIFT** key, the arrows perform tab, back tab, and shift lock functions.

When you press the **UP ARROW**, the cursor jumps to the first line from the second line, then to the top line of the previous screen when pressed again. Backing up will not change entries.

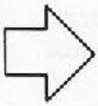
SHIFT plus **UP ARROW** locks the keyboard in uppercase for the current entry.



The **DOWN ARROW** moves the cursor down. The **DOWN ARROW** key enters the displayed answer and moves the cursor to the next line. If there are multiple fields per line, the cursor stays in the same relative column position. If the next line contains only one field, the cursor moves to the beginning of that field. Thus, the **DOWN ARROW** may be used to move quickly through set-ups where most of the default answers are selected.

SHIFT plus **DOWN ARROW** changes the keyboard entry from upper to lowercase.

The **LEFT ARROW** moves the cursor one character to the left until it reaches the beginning of the current field. When the cursor is at the beginning of the second or third field within a line, the **LEFT ARROW** acts as a **BACK TAB,** moving the cursor to the beginning of the previous field. You may also back tab by pressing the **SHIFT** plus **LEFT ARROW** keys. To move quickly through a long text entry, use the **FAST** plus **LEFT ARROW.**



The **RIGHT ARROW** moves the cursor one character at a time to the right until the end of the field. If the cursor is at the end of a field and you press the **RIGHT ARROW** key, the cursor moves to the beginning of the next field. To tab forward, press the **SHIFT** plus **RIGHT ARROW** keys together. To move quickly through a long text entry, use the **FAST** plus **RIGHT ARROW.**

The **TEST** key is used to troubleshoot and calibrate the instrument. Your service representative will advise you when and how to use this key.

> **CAUTION**
>
> **Some tests may erase data or alter module parameters stored in the 983's memory.**

HELP generates assistance messages to clarify input requests, explain errors, and define key functions. Press and hold the **HELP** key to read the message. The original screen is restored when the **HELP** key is released. Examples of the various types of help follow.

The HELP message for an entry request explains more about the entry and how to respond to it. For example, the HELP message for the mode selection screen tells how to select a new mode. The HELP message is:

INC/DEC keys scroll the mode type;
ENTER to select a mode.

Holding down the **HELP** key and pressing another key displays a brief description of the key's function. For example, the HELP message for the **MOTOR ON/OFF** key is:

Manually starts/stops the arm drive.
STATUS Key shows current motor state.

When an error appears in the status line, pressing the **HELP** key displays the error message. For example, the HELP message for "Err 83" displays the following error message:

> **Err 83:** Arm zero offset too large. Check arms (locking pins) and sample.

NOTE

The Appendix contains a table of error codes, and their descriptions.

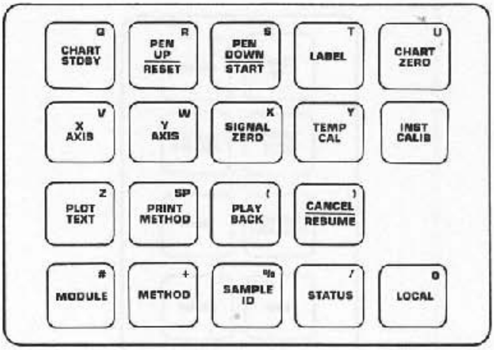


Figure 2.13
Parameter Keys

Seven Parameter keys are used to enter and view information about the experiment. These keys are: **TEMP CAL, INST CALIB, MODULE, METHOD, SAMPLE ID, STATUS,** and **LOCAL.** These keys may be subdivided by function into three categories:

1. Calibration keys are used to calibrate the instrument's performance.
2. Experiment parameter keys permit you to program thermal methods and enter sample identification.
3. Special function keys give status information and remote/local GPIB interface control.

> **NOTE**
> **The plotting and playback keys are not used by the 983 DMA System.**

The keys and their functions are described in Chapter 4.

Operate Keys

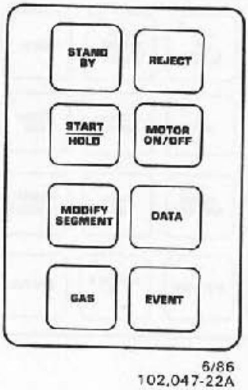


Figure 2.14
Operate Keys

The **OPERATE** keys are used to control the execution of thermal programs. The eight keys start, hold, resume, continue, stop, and reject an experiment, alter a program in progress, control data storage, and set the external event state.

The **STANDBY** key stops the current thermal experiment, discontinues heater power and stops the motor. Data storage is halted, but stored data is retained.

The **REJECT** key cancels the thermal scan in progress and erases the run data. "Reject" is displayed in the status window after the key is pressed. The run number of the rejected run is assigned to the next run. **REJECT** also stops the motor whether or not a method is running.

START / HOLD

The **START** key initiates the thermal program after checking the program method against the mode. It also continues a method from the "Ready" state of an initial temperature segment.

Once the experiment begins, the **HOLD** key suspends the method, maintaining the current thermal and iso-time conditions until the key is pressed again. Data collection and run time are not affected by the **HOLD** key. Press **START** to resume. **HOLD** will not function if the method is in the "set-up" state.

MOTOR ON/OFF

The **MOTOR ON/OFF** key is used to start and stop the motor at any time.

MODIFY SEGMENT

The **MODIFY SEGMENT** key provides a "go to next segment" feature and lets you dynamically alter the segment in progress.

To terminate the current segment execution and begin the next segment, answer **Yes** to the Go To Next Segment question. To modify the parameters for the current segment, answer **No** and the screen will change to the Parameter modification screen. The change is not stored in memory; thus, the program operates as originally created for subsequent runs.

DATA

The **DATA** key provides manual control over data storage. Normally, data collection starts when the program enters the first run state (ramp, isothermal, measure or displace) or when the first Data 1 segment occurs in a program. The **DATA** key may be used to start collection sooner, stop collection in progress, or resume collection. Collection is also controlled by the Data segment in the method.

The GAS key is not used by the 983 DMA.

When it is necessary to synchronize the operation of laboratory equipment, use the EVENT key. You may also override Event segments, 0=off and 1=on, using this key.

> **NOTE**
>
> **To obtain the current data, motor and event states, press the STATUS key.**

CHAPTER 3

Running a Sample Using the 983 System

Running A Sample Using the 983 System

Introduction

This chapter gives a step-by-step description on how to use the 983 Dynamic Mechanical Analyzer. To obtain accurate results with the 983 DMA, follow the procedures carefully and periodically check the calibration of the instrument. After becoming familiar with the basic procedures described below, refer to Chapter 4 for additional technical information.

Once the 983 DMA is installed and checked, you are ready to run an experiment. To run an experiment, use the following steps:

- Select a sample;
- Remove the oven;
- Insert the arm locking pins;
- Mount the sample;
- Remove the arm locking pins;
- Reinstall the oven;
- Set up the general experimental conditions, and
- Start the experiment.

Illustrations can be found throughout this chapter which demonstrate the 983 Dynamic Mechanical Analyzer operating procedures.

General Experiment Conditions

The following procedures describe how to run the 983 DMA in all modes using the polycarbonate sample included in the DMA component accessory kit. These procedures assume the 983 has been calibrated since it is calibrated before shipment, and sent with a list of calibration constants. If the instrument needs to be calibrated or has been recalibrated, a new list of calibration constants should be generated. Calibration constant values shown in the sample runs are default values and will be displayed only if CMOS memory has been lost in the 983 instrument. Compare the calibration constants displayed with the list. If the values differ, reenter the constants from the list or recalibrate the instrument. Periodic recalibration of the instrument is necessary in order to maintain precise measurements and data. Calibration procedures can be found in Chapter 4 of this manual.

When the calibration of the instrument is complete, correctly mount the sample, and set up the general experiment conditions required, such as selecting a particular mode. Once the 983 DMA has been programmed for a particular mode, it will not need to be reprogrammed for each experiment. Rather, the only information that will need to be changed is the sample information and the method, if necessary, to cover the temperature region for a new sample.

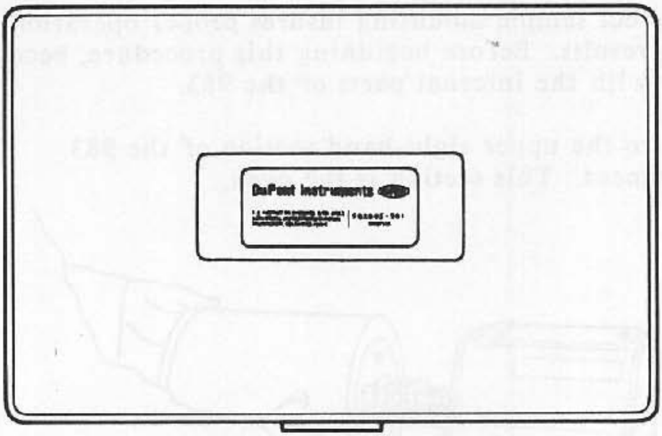


Figure 3.1
983 Component Accessory Kit

983 DMA Component Accessory Kit

Look at the 983 Component Accessory Kit enclosed with the 983. See Figure 3.1. This kit includes:

- 1 Cork Stopper
- 1 1 Amp, Slo Blo Fuse;
- 1 10 Amp, Slo Blo Fuse;
- 1 Torque Wrench;
- 1 Hex Driver for Torque Wrench;
- 1 Open End Wrench;
- 1 1/16 Hex Wrench;
- 1 3/32 Hex Wrench;
- 4 ABS Samples (opaque plastic);
- 4 Polycarbonate Samples (clear);
- 1 Compliance Sample (steel sample for calibration purposes); and
- 1 Thermal Lag Device.
- 2 Constant Tension Springs

These items are important and aid in the calibration and use of the instrument.

Mounting the Sample

Correct sample mounting insures proper operation and accurate results. Before beginning this procedure, become familiar with the internal parts of the 983.

1. Look to the upper right-hand section of the 983 instrument. This section is the oven.

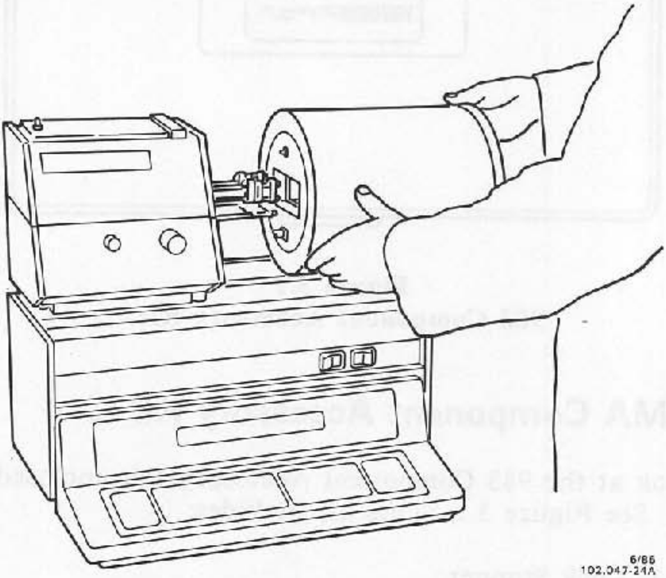


Figure 3.2
983 DMA Oven

2. Locate the two star-shaped knobs on the right end of the oven and loosen them.

3. Carefully, slide the assembly to the right. Using caution, rest it vertically on its protected, black-rimmed end.

4. Focus on the left side of the 983 DMA. This section is called the drive assembly. On the top of the drive assembly is an access cover which has a black knob.

5. Loosen the knob, turning it counter-clockwise, and lift the access cover from the drive assembly.

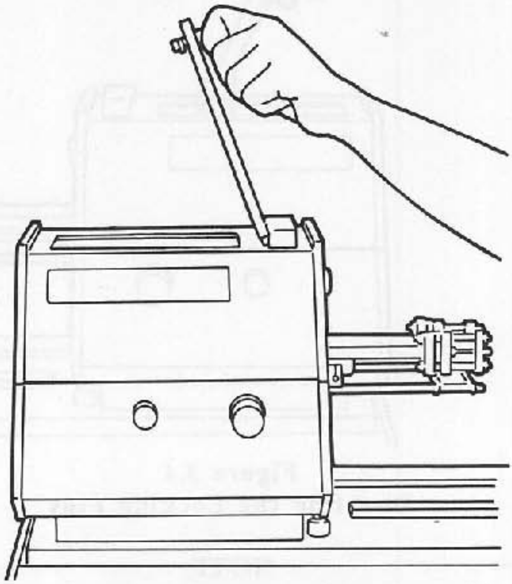


Figure 3.3
983 Drive Assembly

6. Insert the arm locking pins. Locate the two locking pins labeled "front" and "rear." To minimize realignment, place the front pin in the front (balance) arm first, and the rear pin in the rear (driver) arm last. To remove the locking pins from the drive assembly access opening, raise the pin handles to a vertical position and pull straight up. If a locking pin appears to be stuck, it may need adjusting. The instrument is shipped with the locking pins installed. To install the pins, first place the Front Locking Pin in its respective hole with the handle up, pushing down to lock in place. Next, place the Rear Locking Pin in its hole following the same procedure to lock in place.

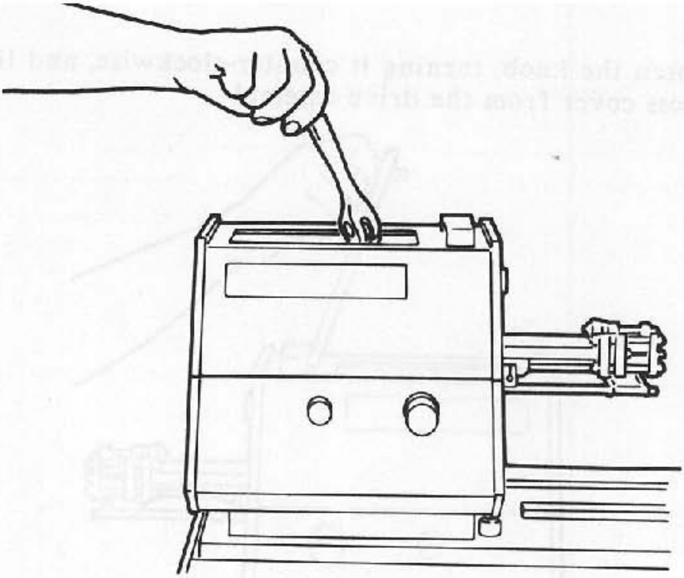


Figure 3.4
Inserting the Locking Pins

> **NOTE**
> **The locking pins prevent damage to the pivots. When installed and locked, they reduce stress to the pivots that occurs when tightening or loosening the clamps while changing samples. Frequently check the locking pins for proper adjustment. Refer to the adjustment procedure in Chapter 5 of this manual.**

7. Remove a mounted sample by using the torque wrench to loosen the clamp screws. Slide the sample from the clamps.

8. Select the experiment sample.

9. Measure the sample width and thickness to ±0.02 mm with a set of calipers, recording all measurements.

CAUTION

Before mounting the sample, make sure locking pins are installed.

10. Slide the sample into the clamps centering it so it is parallel to the base of the instrument. Hand-tighten the clamping screw enough to hold the sample in place.

The following figure shows correctly and incorrectly mounted samples. Be sure the sample is centered between the clamp faces and is not resting on the clamp guide posts.

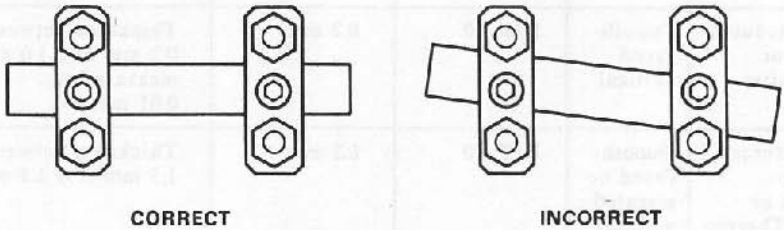


Figure 3.5
Correctly and Incorrectly Mounted Samples

NOTE

Where possible, the sample geometry should be adjusted to obtain a Resonant frequency of approximately 30 Hz. To increase the resonant frequency, increase the sample thickness, and sample width, or decrease the sample length.

The sample mounting procedure described in this section is done with serrated vertical clamps installed on the instrument. To determine the best clamping arrangement for a particular sample, refer to the Sample Mounting Guide found below. To change clamps, turn to the maintenance section (Chapter 5) of this manual.

Table 3.1
Sample Mounting Guide

Sample	Clamp	Sample Dimensions	Oscillation Amplitude	Comments
High Modulus Metals or Composites	Smooth-faced vertical	L/T>10	0.2 mm	Thickness between 0.2 mm and 1.0 mm measured to 0.01 mm.
Unreinforced Thermo-plastics or Cured Thermo sets (below Tg)	Smooth-faced or serrated vertical	L/T>10	0.2 mm	Thickness between 1.5 mm and 3.5 mm.
Brittle solids (ceramics, glasses)	Smooth-faced vertical	L/T>10	0.05 mm to 0.1 mm	Thickness between 0.5 mm and 1.5 mm.
Unreinforced Thermo-plastics or Cured Thermo-sets (below Tg)	Serrated vertical or hori-zontal	L/T=<5	0.2 mm to 0.4 mm	Thickness between 3.5 mm and 15 mm.
Elastomers (above Tg) or Uncured Thermosets	Serrated vertical or hori-zontal	L/T=<5	0.2 mm to 0.4 mm	Thickness between 3.5 mm and 15 mm.

Table 3.1
Sample Mounting Guide (Continued)

Sample	Clamp	Sample Dimensions	Oscillation Amplitude	Comments
Films	Hori-zontal	Length 10 mm to 15 mm	0.1 mm	Film thickness between 0.1 mm and 0.5 mm. (This dimension becomes sample width.)
Glass Cloth supported systems (uncured thermosets, paints, lacquers, prepregs)	Hori-zontal	Length 10 mm to 15 mm	0.1 mm	Ply thickness between 0.2 mm and 0.5 mm. (This dimension becomes sample width.) Modulus values are non-quantitative for this mode of supported system.
Brass Shim Supported Systems (adhesives)	Smooth-faced vertical or hori-zontal	L/T>10	0.2 mm	Thickness between 0.2 mm and 1.0 mm.
Cylindrical Samples (O-rings, tubing, wires)	Notched vertical	--	--	See appropriate sample comments above.

11. Use the torque wrench to tighten the clamp locking screws to 1.1 N m (10 in-lb) for standard samples, 0.3 to 0.6 N m (3 to 5 in-lb) for soft samples, or seven turns after the screw touches the spring on the constant tension springs. See Figure 3.6.

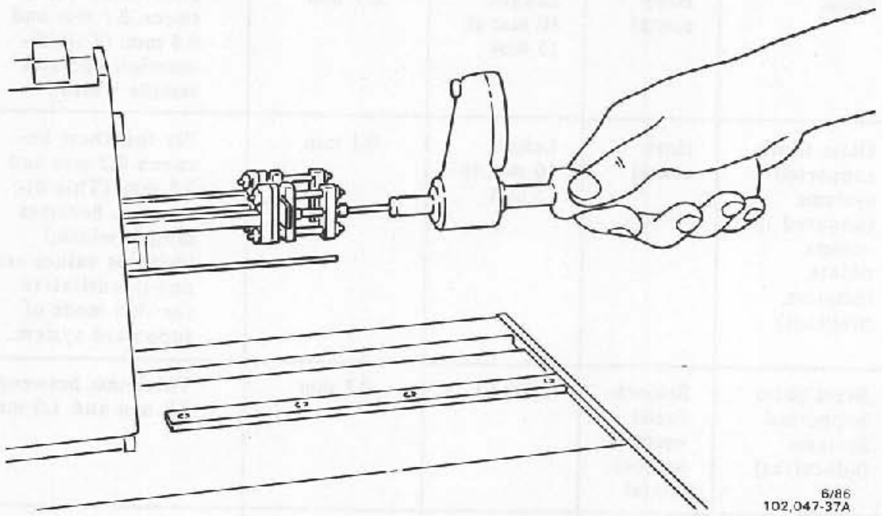


Figure 3.6
View of Tightening the Sample

NOTE

When working with elastomers or soft samples, it is recommended to torque them below their glass transition temperature to avoid sample deformation. An alternative is to load the sample at room temperature and use constant tension spring clamps.

12. Measure the sample length between the clamp faces to ± 0.02 mm with a set of calipers and record the measurement.

13. Check the position of the sample thermocouple. A suitable position is approximately 1 mm away from the sample, one-third the distance from the top of the sample, and one-third the distance from the driven sample arm. The position of the thermocouple can be adjusted using the two setscrews shown in Figure 3.7.

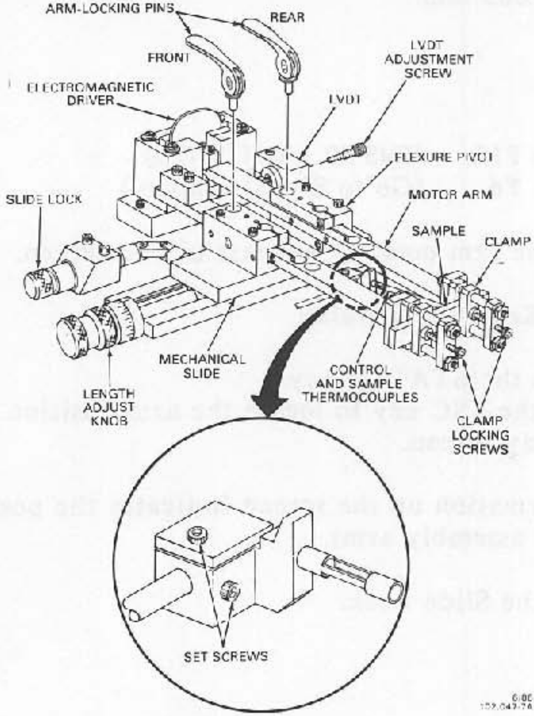


Figure 3.7
View of Sample Arm

> **NOTE**
>
> **A Thermal Lag Device is supplied in the 983 DMA accessory kit. It is used to correct for termal lag in subambient runs with thick samples greater than 5 mm (3/16 in.). Instructions for installing the device appear in Chapter 5.**

14. Position the sample tray under the sample and the radiant shield above the sample.

15. Remove the locking pins and replace the access cover.

16. Slide the oven over the sample. Locate the two star-shaped knobs on the right of the oven and tighten them.

17. If the arms are not at zero, loosen the Slide Lock and then turn the Length Adjust Knob until the arm position reads ± 0.005 mm.

On the 9900

A. Press **F12.** (INSTR CONTROL)
B. Press **F6.** (Go to Signal Control)

- The arm position appears on the screen.

On the Local Keyboard/Display

A. Press the **STATUS** key.
B. Use the **INC** key to locate the arm position on the display screen.

The information on the screen indicates the position of the drive assembly arms.

18. Tighten the Slide Lock.

9900 Procedures

Initial Operating Procedures

When using the 983 DMA with the 9900, both instruments should be installed properly. Before setting up an experiment, make sure you have:

- Made all of the necessary cable connections from the 983 DMA to the 9900;
- Powered on each unit;
- Installed all available options;
- Set up the system with available formatted disks; and,
- Become familiar with the keyboard.

If all the above steps have been performed, you are ready to use the 9900 with the 983 DMA.

NOTE

The instructions, procedures,and examples, outlined in this chapter assume the 9900 is correctly connected to the 983 DMA.

To run a experiment, have all samples and equipment necessary to perform the experiment. Ensure that the 983 DMA is connected properly to the 9900 and is operational. Mount the sample in the 983 DMA module as directed in the beginning of this chapter. Let's begin.

Step 1: Press **F12**, INSTR CONTROL.

> **NOTE**
>
> **If there are no modules on line, press F8, GoTo Configure System, and then press F5, Auto Configure. After the DMA is configured, press F12 again.**

- The following display screen appears.

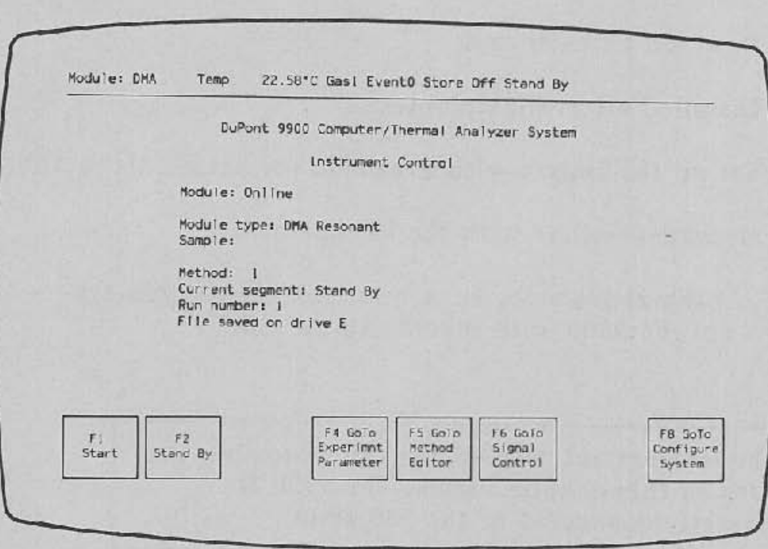


Figure 3.8
Instrument Control Start-up Screen

The screen automatically displays the status of an experiment. Enter the information concerning the 983 DMA module on the Experimental Parameters screen. Upon returning to the Instrument Control screen, all the appropriate information is displayed.

A row of boxes appears at the bottom of the Instrument Control screen. These boxes represent the function keys. The table on the following page lists and briefly defines each function key.

Table 3.2
Instrument Control Function Keys

Function Key	Definition
F1 Start	Starts the method that is displayed.
F2 Stand By	Puts the module in the standby mode, stopping the method and the motor drive.
F4 GoTo Experiment Parameters	Takes you to the Experimental Parameters screen, permitting the method selection, identification of the sample, and the mode selection.
F5 GoTo Method Editor	Retrieves the Method Editor screen, which is used to create, change, copy, or delete a method.
F6 GoTo Signal Control	Goes to the Signal Control screen which displays the module signals, and controls special module functions.
F8 GoTo Configure System	Returns to the System Configuration screen.

NOTE

If you press the wrong key or want to return to the previous display, press the ESCAPE key. F12 will always display the Instrument Control screen and F11 will always display the Data Analysis screen.

Step 2: Press **F4**. The Experimental Parameters screen appears.

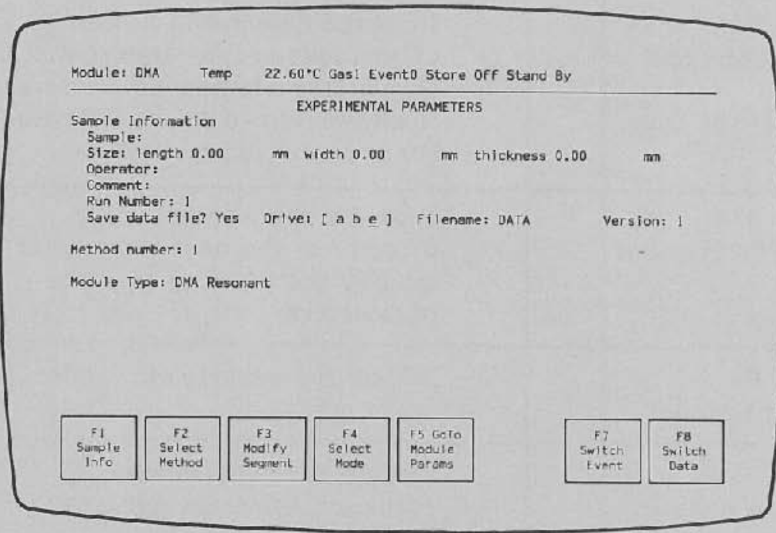


Figure 3.9
Experimental Parameters Screen

Notice the seven function keys at the bottom of this screen. Each of these keys aids in entering necessary experiment data. The following table defines the primary function of each key.

Table 3.3
Experimental Parameter Function Keys

Function Key	Definition
F1 Sample Info	Enters data in the sample information section of the screen.
F2 Select Method	Lists the names and numbers of methods previously created by way of the Method Editor screen. It allows you to select a method for the next experiment.
F3 Modify Segment	Provides the means to skip directly to the next segment or modify the running segment parameters.
F4 Select Mode	Selects the experiment mode.
F5 GoTo Module Params	Sets up the experimental parameters for the selected instrument mode.
F7 Switch Event	Toggles on and off the event relay.
F8 Switch Data	Toggles on and off the store function that functions only when a method is running.

Resonant Mode

The following information describes a polycarbonate sample being run in the resonant mode from the 9900. Once acquainted with the basic function of the 9900, set up an experiment using the following directions.

Step 1: Selecting the Instrument Mode

A. Press **F12**, Instrument Control.

- If the module has not been configured, see the NOTE on page 3-14.

B. Press **F4**, Go to Experimental Parameters.

- Before running a mode, the 9900 must know something about it. Enter mode data by way of the Function keys, displayed on the Experimental Parameters screen.

C. Press **F4**, Select Mode.

- The following Mode Selection screen appears.

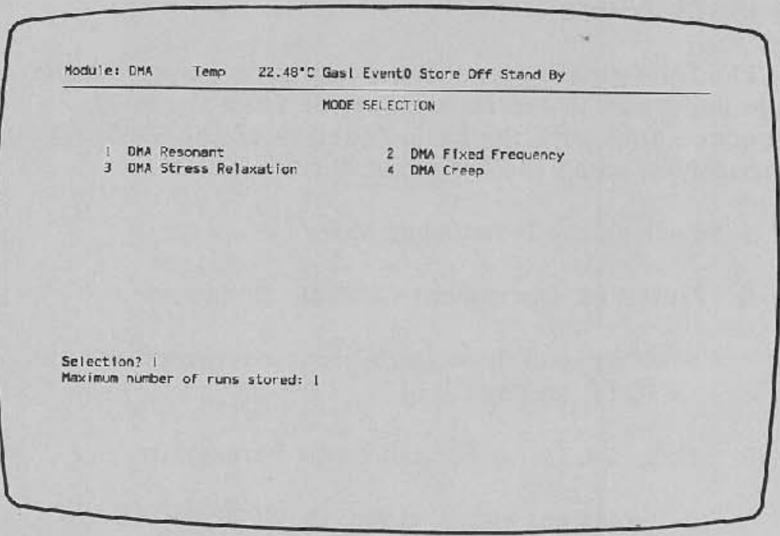


Figure 3.10
Mode Selection Screen

D. Type **1** and press **RETURN.**

- The **RETURN** key automatically moves to the next input prompt. "Maximum number of runs stored:"

E. Type **1** and press **RETURN.**

- Once the number is entered, the Experimental Parameters screen appears.

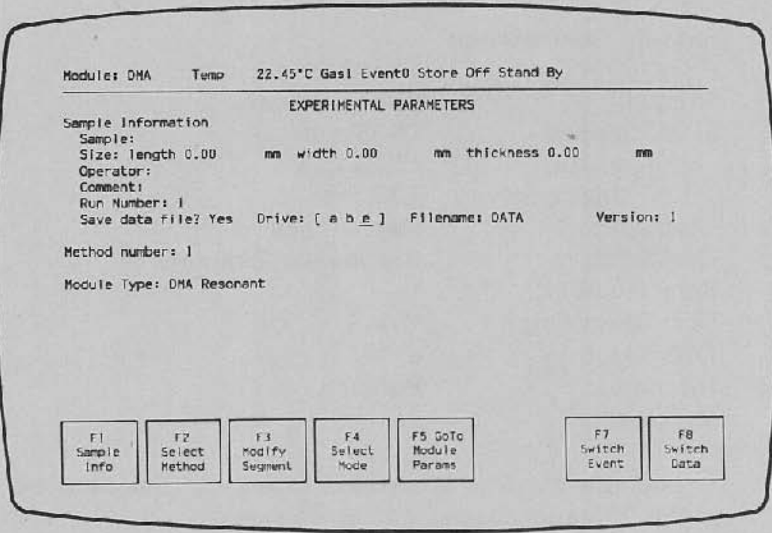


Figure 3.11
Experimental Parameters Screen

Step 2: Entering Sample Information

A. Press **F1**, Sample Information.

- To run an experiment in the resonant mode, input the required information.

B. Complete each of the following:

- Press **RETURN** as each field is completed.
- The cursor automatically moves to the next field.

Sample Information

Sample:	**Polycarbonate**
Size: length:	**25.35** mm
width:	**12.78** mm
thickness:	**3.12** mm
Operator:	**Super Tech**
Comment:	**Resonance Example**
Run Number:	*
Save Data File?	**Yes**
Drive [a b e]:	**e**
Filename:	**Polcarb**
Version:	**1**

* The run number is automatically incremented by the 983 and should not be changed.

- At the bottom of the screen the following information appears.

Method Number: 1
Module Type: DMA Resonant

Step 3: Selecting the Method

A. Press **F2, Select** Method.

- The Method Selection screen appears.

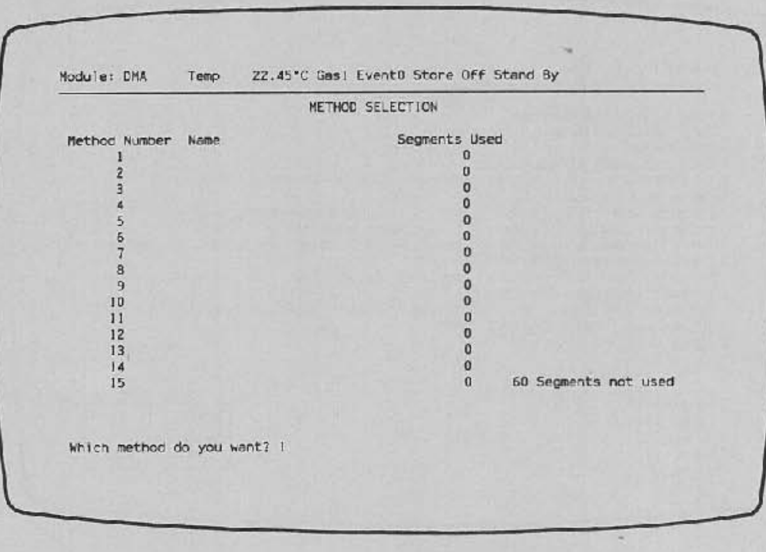


Figure 3.12
Method Selection Screen

This particular screen lists each of the 15 methods available in the system. Select the desired method number. This example uses method 1.

B. Type **1**, and press **RETURN**.

Step 4: Inputting the Module Parameters

A. Press **F5**, Module Parameters.

- The Module Parameters screen appears.

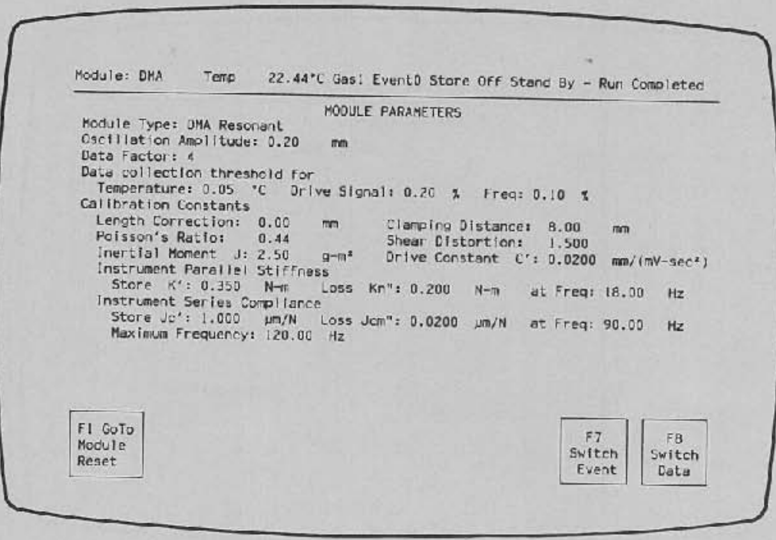


Figure 3.13
Module Parameters Screen

B. Enter the Module Parameters completing the necessary information requested on this display.

NOTE

The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

Module Type: (**DMA Resonant** will be displayed.)
Oscillation Amplitude: 0.20 mm
Data Factor: 4

Data Collection threshold for:	
Temperature:	0.05°C
Drive Signal:	0.20 %
Freq:	0.10 %
Calibration constants	
Length Correction:	0.00 mm
Clamping Distance:	8.00 mm
Poission's Ratio:	0.44
Shear Distortion:	1.500
Inertial Moment:	2.50 $g\text{-}m^2$
Drive Constant:	0.0200 $mm/(mV\text{-}sec^2)$
Instrument Parallel Stiffness:	
Store:	0.350 N-m
Loss:	0.200 N-m
at Freq:	18.00 Hz
Instrument Series Compliance	
Store:	1.000 μm/N
Loss:	0.020 μm/N
at Freq:	90.00 Hz
Maximum Frequency:	120.0 Hz

C. If the existing entry is correct press **RETURN**, otherwise enter a new value. The cursor automatically moves to the next field.

D. Press **RETURN** after the last entry on the screen to accept all values.

You have completed setting up the mode parameters and sample information.

Step 5: Creating the Method

A. Press **F12**, Instrument Control.

B. Press **F5**, Method Editor.

- The Method Editor screen appears.

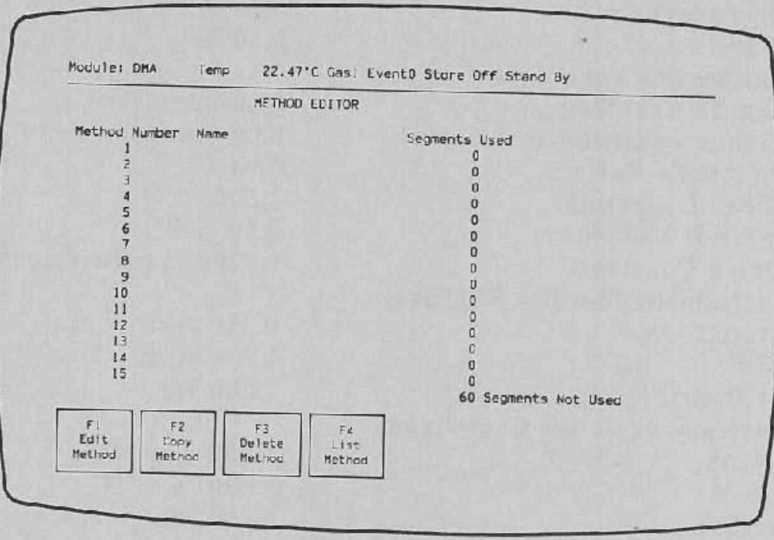


Figure 3.14
Method Editor Screen

C. Press **F1** to edit a method.

- Select a method number; this example uses 1.

D. Type the method number and press **RETURN**. Next, complete the method description.

E. Method Name: **5°C/min Ramp**

- Type in the applicable response and press **RETURN**.

NOTE

After the RETURN key is pressed, more questions will appear on the screen.

F. Create the following method:

1 Equilibrate at **100.00°C**
2 Ramp **5.00°C**/min to **190.00°C**

G. Press **F6**, Accept Method.

- This key stores all the method information in the 983 DMA module.

Step 6: Running the Experiment.

A. Press **F12** to return to the Instrument Control screen.

- The Instrument Control screen is complete. Now, run an experiment using the 9900 with the 983 DMA.

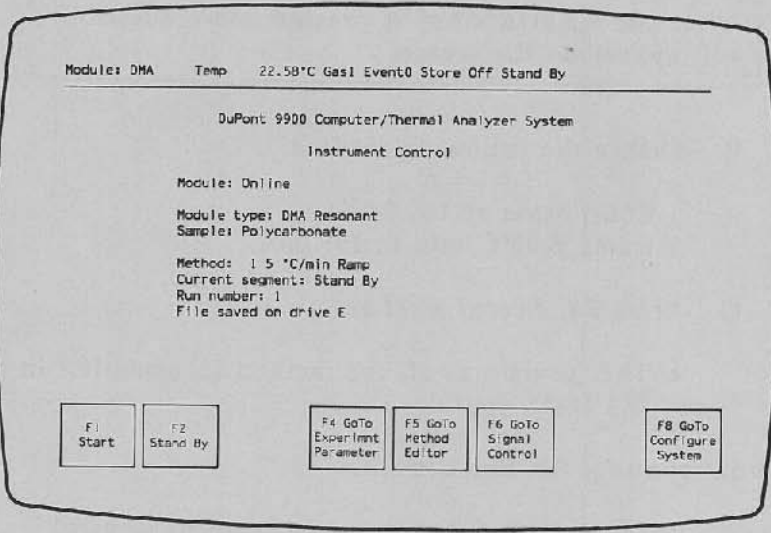


Figure 3.15
Completed Instrument Control Screen

B. Check the 983 DMA module to see if the sample is in place, the oven is installed, and the **HEATER** switch is turned on.

C. Press **F1**, the Start key to begin the experiment.

D. Press **F6**, Signal Control, to monitor the experiment.

- Real-time plot can be used to plot raw data in real time.

9900 Procedures

Fixed Frequency Mode

The following information describes a polycarbonate sample being run in the fixed frequency mode from the 9900. Once acquainted with the basic function of the 9900, set up an experiment using the following directions.

Step 1: Selecting the Instrument Mode

A. Press **F12**, Instrument Control.

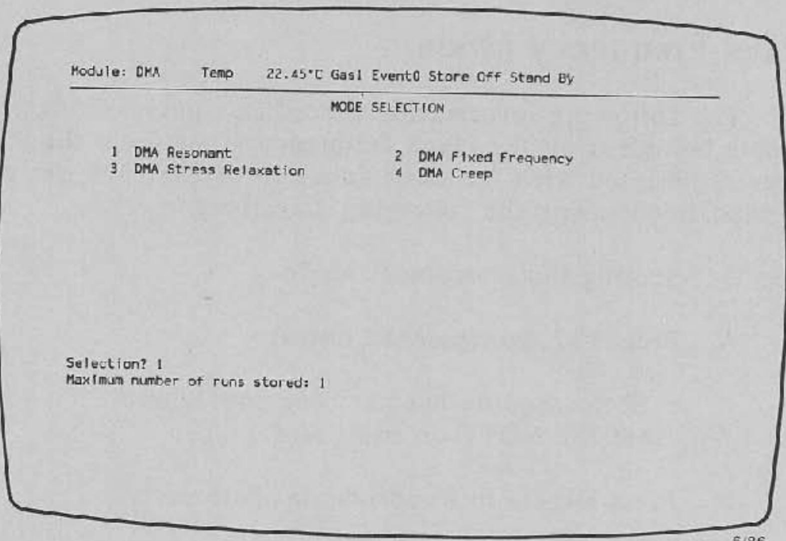
- If the module has not been configured, see the NOTE on page 3-14.

B. Press **F4**, Go to Experimental Parameters.

- Before running a mode, the 9900 must know something about it. Enter mode data by way of the Function keys displayed on the Experimental Parameters screen.

C. Press **F4**, Select Mode.

- The Mode Selection screen appears.



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102.047-17A

Figure 3.16
Mode Selection Screen

D. Type 2, and press **RETURN**.

- The **RETURN** key automatically moves to the next input prompt. "Maximum number of runs stored:"

E. Type 1 and press **RETURN**.

- Once the number is entered, the Experimental Parameters screen appears.

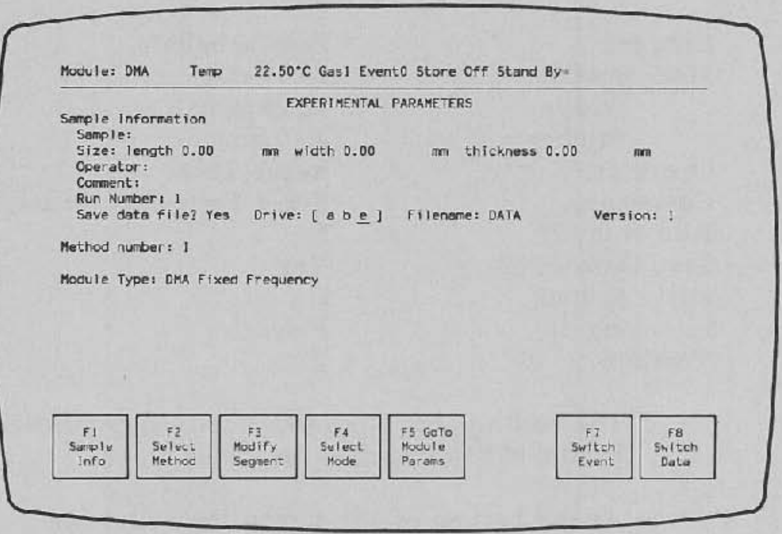

Figure 3.17
Experimental Parameters Screen

Step 2: Entering Sample Information

A. Press **F1**, Sample Information.

- In order to run an experiment in the fixed frequency mode, input the required information.

B. Complete each of the following:

- Press **RETURN** as each entry is completed.

- The cursor automatically moves to the next field.

Sample Information

Sample:	**Polycarbonate**
Size: length:	**25.35 mm**
width:	**12.78 mm**
thickness:	**3.12 mm**
Operator:	**Super Tech**
Comment:	**Fixed Frequency Example**
Run Number:	*
Save Data File?	**Yes**
Drive [a b e]:	**e**
Filename:	**Polcarb**
Version:	**2**

* The run number is automatically incremented by the 983 and should not be changed.

- At the bottom of the screen the following information appears.

Method Number: 1
Module Type: DMA Fixed Frequency

Step 3: Selecting the Method

A. Press **F2**, Select Method.

- The Method Selection screen appears.

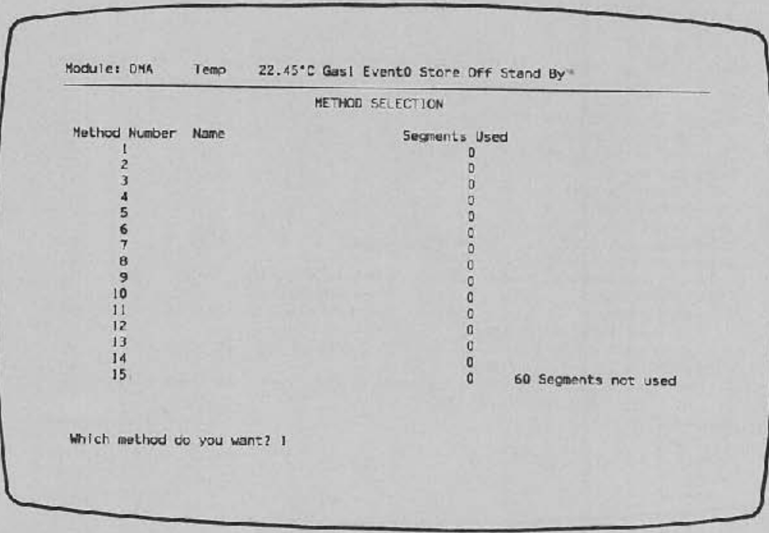

Figure 3.18
Method Selection Screen

This particular screen lists each of the 15 methods available in the system. Select the desired method number. This example uses method 1.

B. Type 1, and press **RETURN**.

Step 4: Inputting Module Parameters

A. Press **F5**, Module Parameters.

- The Module Parameter screen appears.

```
Module: DMA      Temp    22.45°C Gas1 Event0 Store Off Stand By - Run Completed
                              MODULE PARAMETERS
Module Type: DMA Fixed Frequency
Frequency: 1.000  Hz
Oscillation Amplitude: 0.50    mm
Data Factor: 4
Equilibrate Cycles: 5
Measurement Points: 5
Data collection threshold for
  Temperature: 0.05  °C   Drive Signal: 0.20  %   Phase: 0.10  %
Calibration Constants
  Length Correction:  0.00    mm       Clamping Distance:  8.00    mm
  Poisson's Ratio:    0.44             Shear Distortion:   1.500
  Inertial Moment  J: 2.50    g-m²     Drive Constant  C': 0.0200  mm/(mV-sec²)
  Instrument Parallel Stiffness
    Store  K': 0.350   N-m    Loss  Kn": 0.200   N-m    at Freq: 18.00   Hz
  Instrument Series Compliance
    Store Jc': 1.000   µm/N   Loss Jcm": 0.0200  µm/N   at Freq: 90.00   Hz
    Maximum Frequency: 120.00  Hz

F1 GoTo                                                  F7        F8
Module                                                 Switch    Switch
Reset                                                  Event      Data
```

Figure 3.19
Module Parameters Screen

B. Enter the Module Parameters, completing the information requested on this display.

NOTE

The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

Module Type: (**DMA Fixed Frequency** will be displayed.)

Frequency: **1.00** Hz

- This is the value which will be used in the Fixed Frequency mode to run the experiment.

Oscillation Amplitude: **0.50** mm

Data Factor:	**4**
Equilibrate Cycles:	**5**
Measurement Points:	**5**
Data Collection threshold for	
Temperature:	**0.05°C**
Drive Signal:	**0.20 %**
Phase:	**0.10 %**
Calibration Constants	
Length Correction:	**0.00** mm
Clamping Distance:	**8.00** mm
Poission's Ratio:	**0.44**
Shear Distortion:	**1.500**
Inertial Moment:	**2.50** g-m^2
Drive Constant:	**0.0200** mm/(mV-sec^2)
Instrument Parallel Stiffness:	
Store:	**0.350** N-m
Loss:	**0.200** N-m
at Freq:	**18.00** Hz
Instrument Series Compliance	
Store:	**1.000** μm/N
Loss:	**0.020** μm/N
at Freq:	**90.00** Hz
Maximum Frequency:	**120.0** Hz

C. If the existing entry is correct press **RETURN**, otherwise, enter a new value. The cursor automatically moves to the next field.

D. Press **RETURN** after the last entry on the screen to accept all values.

You have completed setting up the mode parameters and sample information.

Step 5: Creating a Method

A. Press **F12**, Instrument Control.

B. Press **F5**, Method Editor.

- The Method Editor screen appears.

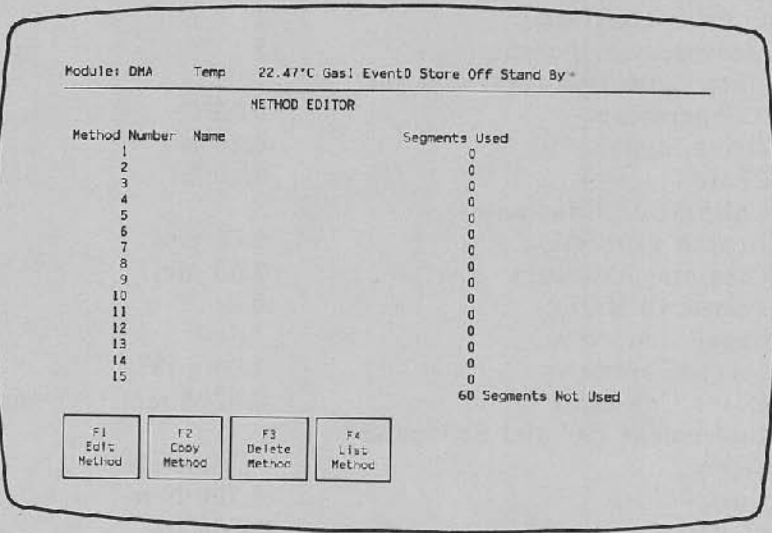

Figure 3.20
Method Editor Screen

C. Press **F1** to edit a method.

- Select a method number; this example uses 1.

D. Type the method number and press **RETURN**. Next, complete the method description.

E. Method Name: **5°C/min Ramp**

- Type in the applicable response and press **RETURN**.

NOTE

As the RETURN key is pressed, more questions will appear on the screen.

F. Create this following method:

1 Equilibrate at **100.00°C**
2 Ramp **5.00°C/min** to **190.00°C**

G. Press **F6**, Accept Method.

- This key stores all of the method information in the 983 DMA module.

Step 6: Running the Experiment

A. Press **F12** to return to the Instrument Control screen.

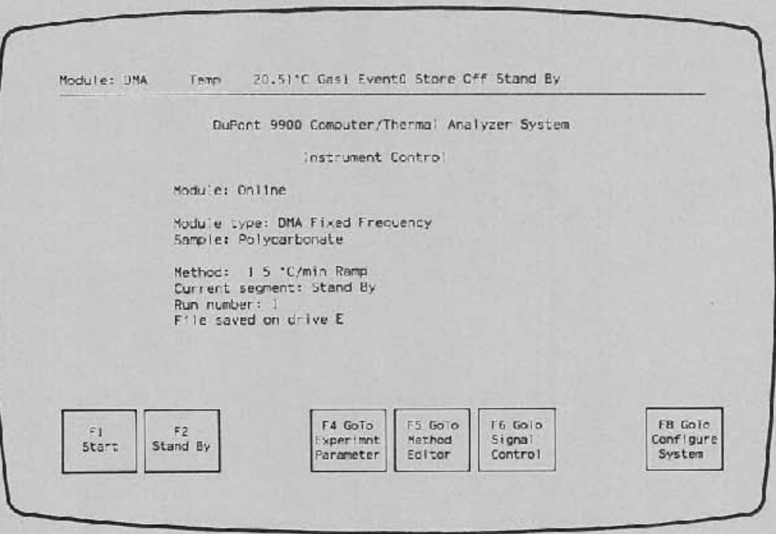

Figure 3.21
Completed Instrument Control Screen

- After the Instrument Control screen is complete, the 983 DMA is ready to run an experiment using the 9900.

B. Check the 983 DMA module to see if the sample is in place, the oven is installed, and the **HEATER** switch is turned on.

C. Press **F1**, the Start key, to begin the experiment.

D. Press **F6**, the Signal Control key, to monitor the experiment.

  - Real-time plot can be used to plot raw data in real time.

Multiple Fixed Frequencies

When running an experiment with multiple fixed frequencies, note that its experimental set-up varies somewhat from the fixed frequency mode. Follow this procedure.

Step 1: Set up the instrument in the fixed frequency mode. Refer to the fixed frequency experiments section for assistance.

Step 2: Creating the method.

A. Press **F12**, Instrument Control.

B. Press **F5**, Method Editor.

C. Select the method number.

- This example uses 2.

D. Type in the number and press **RETURN**. Next, complete the method description.

E. Method Name: **Multiple Frequencies**

- Type in the applicable response and press **RETURN**.

F. Create the following method:

Method Segments

1. Equilibrate at **130.00°C**
2. Measure at frequency **0.250** Hz
3. Measure at frequency **0.500** Hz
4. Measure at frequency **1.000** Hz
5. Measure at frequency **2.000** Hz
6. Increment **2.00°C**
7. Repeat segment **2** til **160.00°C**

G. Press **F6**, Accept Method.

- This key accepts the method information and enters the data.

Step 3: Running the experiment.

Run the experiment as described in the fixed frequency mode.

Stress Relaxation Mode

The following information describes a polycarbonate sample being run in the stress relaxation mode from the 9900. Once acquainted with the basic function of the 9900, set up an experiment using the following directions.

Step 1: Selecting the Instrument Mode

A. Press **F12**, Instrument Control.

- If the module has not been configured, see the NOTE on page 3-14.

B. Press **F4**, Go to Experimental Parameters.

- Before running a mode, the 9900 must know something about it. Enter mode data by way of the Function keys, displayed on the Experimental Parameters screen.

C. Press **F4**, Select Mode.

- The Mode Selection screen appears.

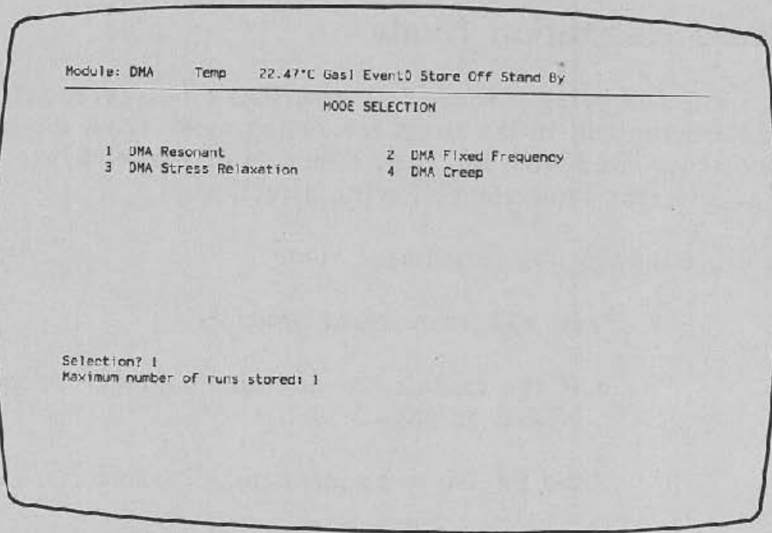

Figure 3.22
Mode Selection Screen

D. Type 3, and press **RETURN**.

- The **RETURN** key automatically moves to the next input prompt. "Maximum number of runs stored:"

E. Type **1** and press **RETURN**.

- Once the number is entered, the Experimental Parameters screen appears.

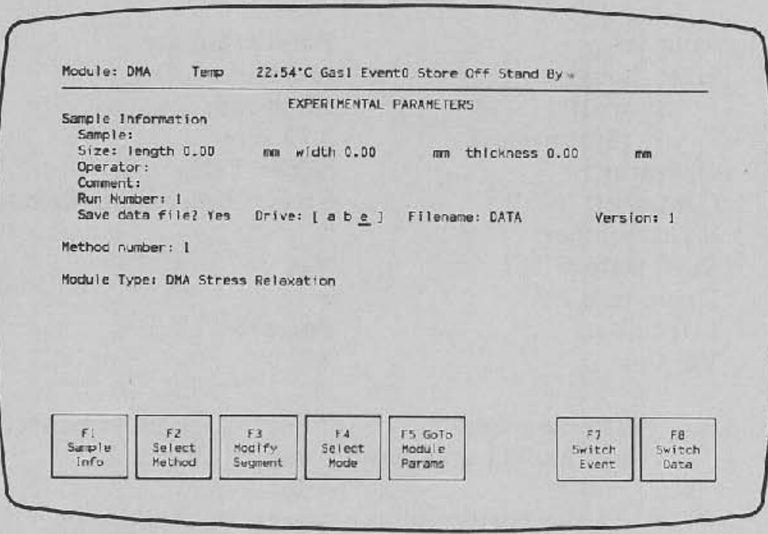

Figure 3.23
Experimental Parameters Screen

Step 2: Entering Sample Information

A. Press **F1**, Sample Information.

- To run an experiment in the stress relaxation mode, input the required information.

B. Complete each of the following:

- Press **RETURN** as you complete each answer.
- The cursor automatically moves to the next field.

Sample Information

Sample:	**Polycarbonate**
Size: length:	**25.35 mm**
width:	**12.78 mm**
thickness:	**3.12 mm**
Operator:	**Super Tech**
Comment:	**Stress Relaxation Example**
Run Number:	*
Save Data File?	**Yes**
Drive [a b e]:	**e**
Filename:	**Polcarb**
Version:	**3**

* The run number is automatically incremented by the 983 and should not be changed.

- At the bottom of the screen the following information appears.

Method Number: 1
Module Type: DMA Stress Relaxation

Step 3: Selecting the Method

A. Press **F2**, Select Method.

- The Method Selection screen appears:

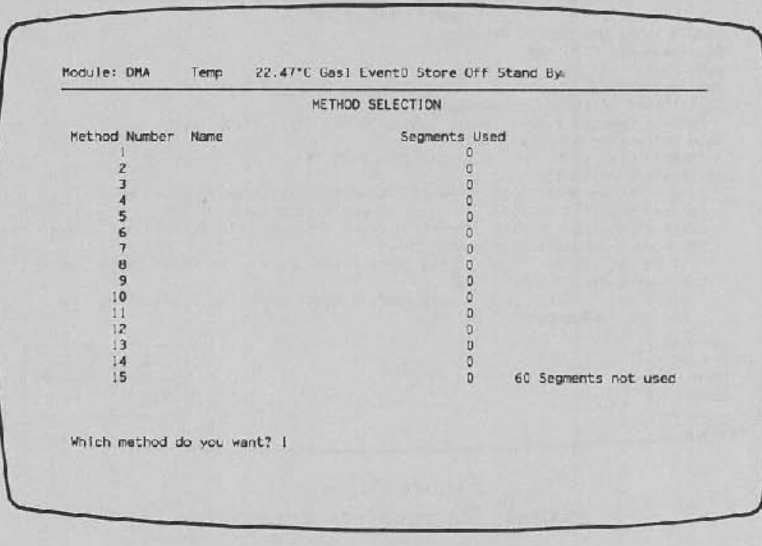

Figure 3.24
Method Selection Screen

This particular screen lists each of the 15 methods available in the system. Select the desired method. This example uses method 3.

B. Type **3**, and press **RETURN**.

Step 4: Inputting the Module Parameters

A. Press **F5**, Module Parameters.

- The Module Parameter screen appears.

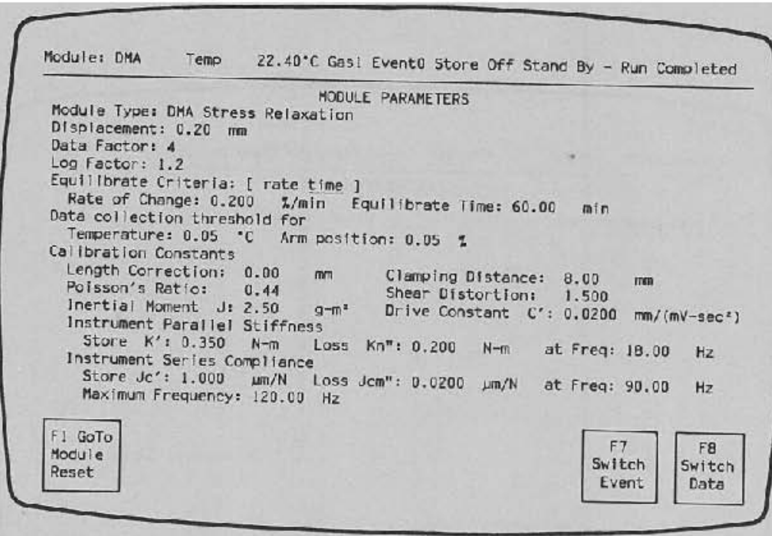

Figure 3.25
Module Parameters Screen

B. Enter the Module Parameters, completing the requested information.

NOTE

The following values are typical values and should be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

Module Type: (**DMA Stress Relaxation** will be displayed.)

Displacement:	**0.50 mm**
Data Factor:	**4**
Log Factor:	**1.2**
Equilibrate Criteria: [rate <u>time</u>]	
Rate of Change:	**0.200 %/min**
Equilibrate Time:	**60.00 min**

Data Collection threshold for

Temperature:	**0.05°C**
Arm position	**0.05 %**

Calibration Constants	
Length Correction:	**0.00** mm
Clamping Distance:	**8.00** mm
Poisson's Ratio:	**0.44**
Drive Constant:	**0.0200** mm/(mV-sec^2)
Shear Distortion:	**1.500**
Inertial Moment:	**2.50** g-m^2
Instrument Parallel Stiffness	
Store:	**0.350** N-m
Loss:	**0.200** N-m
at Freq:	**18.00** Hz
Instrument Series Compliance	
Store:	**1.000** μm/N
Loss:	**0.020** μm/N
at Freq:	**90.00** Hz
Maximum Frequency:	**120.0** Hz

C. If the existing entry is correct press **RETURN**, otherwise enter a new value. The cursor automatically moves to the next field.

D. Press **RETURN** after the last entry on the screen to accept all values.

- You have completed setting up the mode parameters and sample information.

Step 5: Creating the Method

A. Press **F12**, Instrument Control.

B. Press **F5**, Method Editor.

- The Method Editor screen appears.

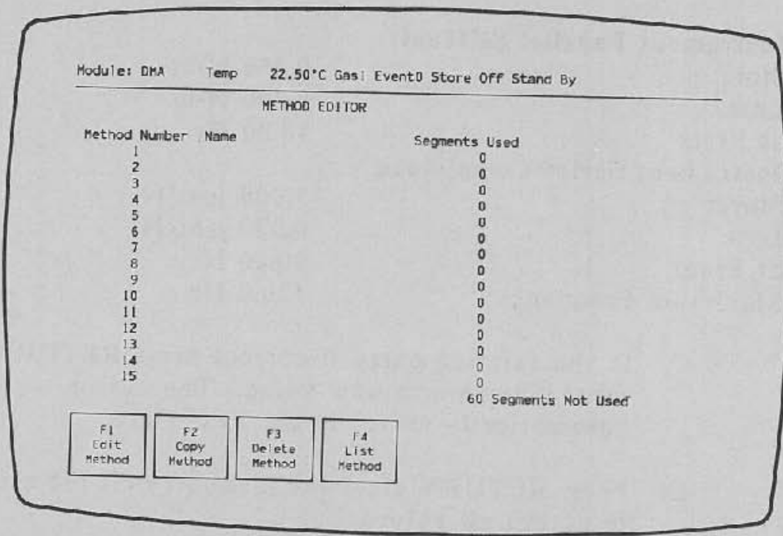

Figure 3.26
Method Editor Screen

C. Press **F1** to edit a method.

- Select a method number. This example uses 3.

D. Type the method number and press **RETURN**. Next, complete the method description.

E. Method Name: **10°C Inc & Displace.**

- Type in the applicable response and press **RETURN**.

NOTE

After the RETURN key is pressed more questions will appear on the screen.

F. Create the following method.

1 Equilibrate at **60.00°C**
2 Displace **100.00** min Recover **0.00** min
3 Increment **10.00°C**
4 Repeat segment 2 til **140.00°C**

G. Press **F6**, Accept Method.

- This key stores all of the method information in the 983 DMA module.

Step 6: Running the Experiment

A. Press **F12** to return to the Instrument Control screen.

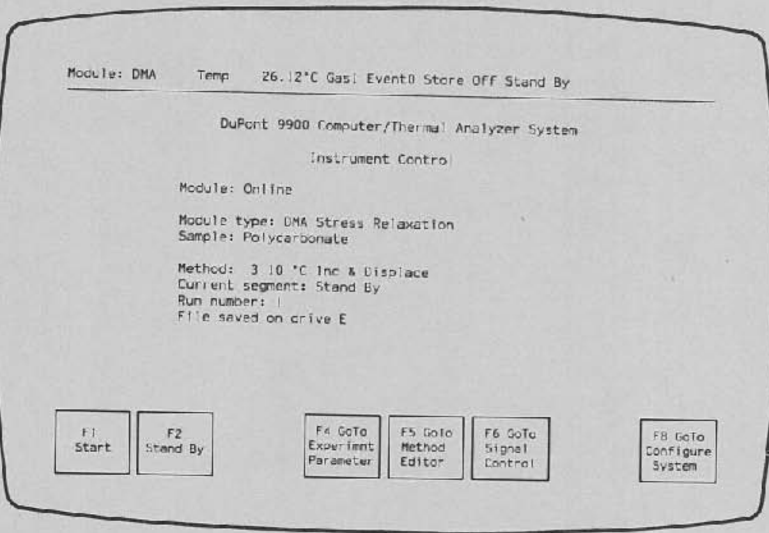

Figure 3.27
Completed Instrument Control Screen

- The Instrument Control screen is complete. Now the 983 DMA is ready to run an experiment using the 9900.

B. Check the 983 DMA module to see if the sample is in place, the oven is installed, and the **HEATER** switch is turned on.

C. Press **F1**, the Start key, to begin the experiment.

D. Press **F6**, the Signal Control key, to monitor the experiment.

- Real-time plot can be used to plot raw data in real time.

9900 Procedures

Creep Mode

The following information describes a polycarbonate sample being run in the creep mode from the 9900. Once acquainted with the basic function of the 9900, begin setting up an experiment using the following directions.

Step 1: Selecting the Instrument Mode

A. Press **F12**, Instrument Control.

- If the module has not been configured, see the NOTE on page 3-14.

B. Press **F4**, Go to Experimental Parameters.

- Before running a mode, the 9900 must know something about it. Enter mode data by way of the Function keys displayed on the Experimental Parameters screen.

C. Press **F4**, Select Mode.

- The Mode Selection screen appears.

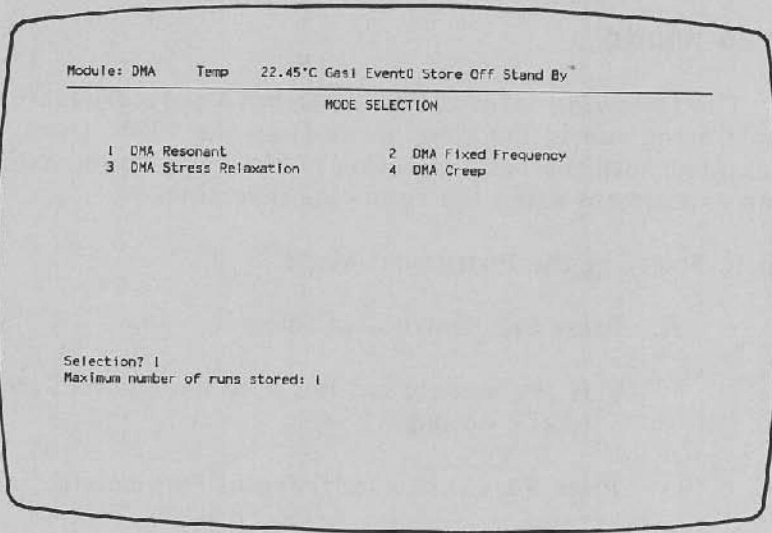

Figure 3.28
Mode Selection Screen

D. Type 4, and press **RETURN**.

- The **RETURN** key automatically moves to the next input prompt. "Maximum number of runs stored:"

E. Type 1 and press **RETURN**.

- Once the number is entered, the following Experimental Parameters screen appears.

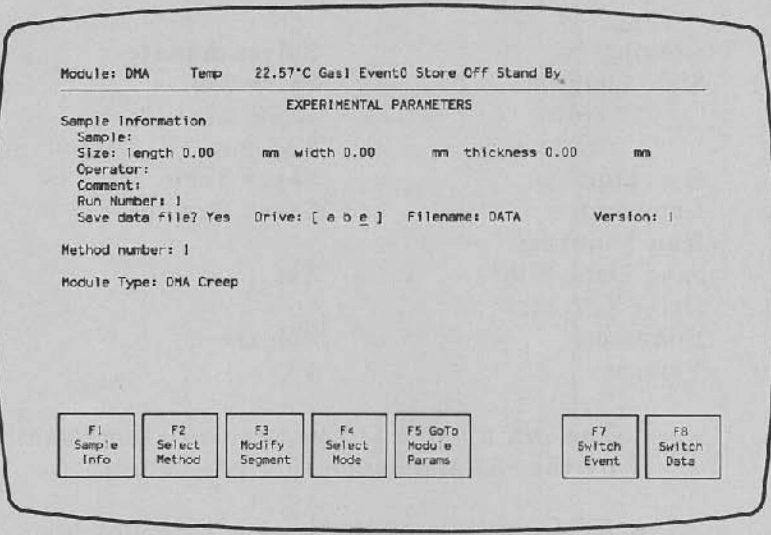

Figure 3.29
Experimental Parameters Screen

Step 2: Enter Sample Information.

A. Press **F1**, Sample Information.

- To run an experiment in the creep mode, input the required information.

B. Complete each of the following:

- Press **RETURN** as each entry is completed.
- The cursor automatically moves to the next field.

Sample Information

Sample:	**Polycarbonate**
Size: length:	**25.35** mm
width:	**12.78** mm
thickness:	**3.12** mm
Operator:	**Super Tech**
Comment:	**Creep Example**
Run Number:	*
Save Data File?	**Yes**
Drive [a b e]:	**e**
Filename:	**Polcarb**
Version:	**4**

* The run number is automatically incremented by the 983 and should not be changed.

- At the bottom of the screen the following information appears.

Method Number: 1
Module Type: DMA Creep

Step 3: Selecting the Method

A. Press **F2**, Select Method.

- The Method Selection screen appears:

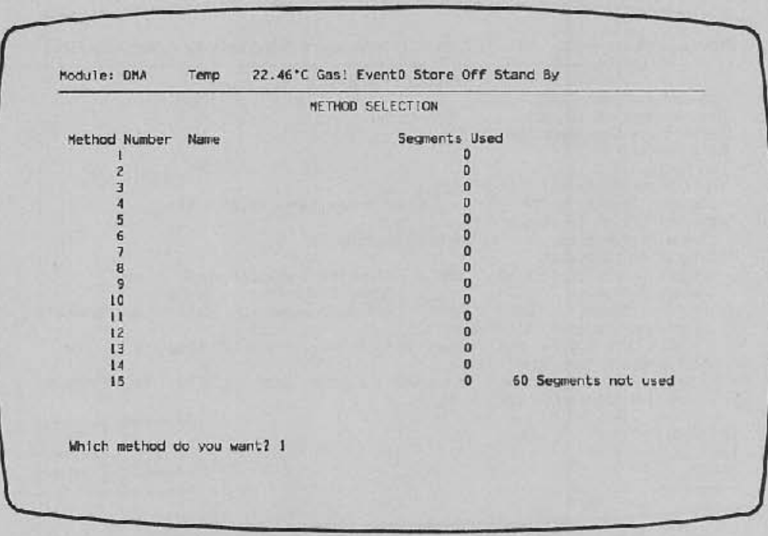

Figure 3.30
Method Selection Screen

- This particular screen lists each of the 15 methods available in the system. Select the desired method. This example uses method 3.

B. Type **3**, and press **RETURN**.

Step 4: Inputting the Module Parameters

A. Press **F5**, Module Parameters.

- The Module Parameters screen appears.

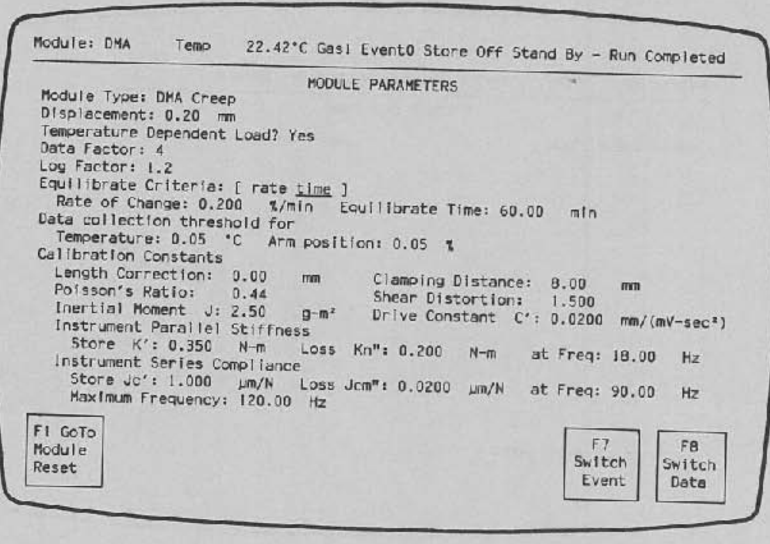

Figure 3.31
Module Parameters Screen

B. Enter the Module Parameters by completing the requested information on the display.

NOTE

The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

Module Type: (**DMA Creep** will be displayed.)

Displacement:	0.50 mm
Data Factor:	4
Log Factor:	1.2

Equilibrate Criteria [rate <u>time</u>]

Rate of Change:	0.200 %/min
Equilibrate Time:	60.00 min
Data Collection threshold for	
Temperature:	0.05°C
Arm position :	0.05 %
Calibration Constants	
Length Correction:	0.00 mm
Clamping Distance:	8.00 mm
Poission's Ratio:	0.44
Shear Distortion:	1.500
Inertial Moment:	2.50 g-m^2
Drive Constant:	0.0200 mm/(mV-sec^2)
Instrument Parallel Stiffness	
Store:	0.350 N-m
Loss:	0.200 N-m
at Freq:	18.00 Hz
Instrument Series Compliance	
Store:	1.000 μm/N
Loss:	0.020 μm/N
at Freq:	90.00 Hz
Maximum Frequency:	120.0 Hz

C. If the existing entry is correct press **RETURN**, otherwise enter a new value. The cursor automatically moves to the next field.

D. Press **RETURN** after the last entry on the screen to accept all values.

You have completed setting up the mode parameters and sample information.

Step 5: Creating the Method

A. Press **F12**, Instrument Control.

B. Press **F5**, Method Editor.

- The Method Editor screen appears.

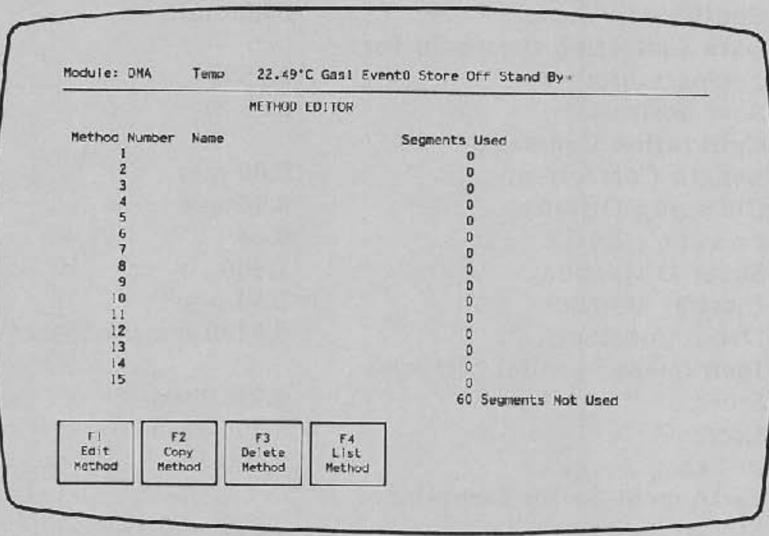

Figure 3.32
Method Editor Screen

C. Press **F1** to edit a method.

- Select the method number. This example uses 3.

D. Type the method number and press **RETURN**. Next, complete the method description.

E. Method Name: **10°C Inc & Displace.**

- Type in the applicable response and press **RETURN**.

NOTE

When RETURN is pressed, more questions will appear on the screen.

F. Create the following method:

1 Equilibrate at **60.00°C**
2 Displace **100.00** min Recover **0.00** min
3 Increment **10.0°C**
4 Repeat segment **2** til **140.00°C**

G. Press **F6**, Accept Method.

- This key stores all of the method information in the 983 DMA module.

Step 6: Running the Experiment

A. Press **F12** to return to the Instrument Control screen.

- The Instrument Control screen is complete. Now, run an experiment using the 9900 with the 983 DMA.

B. Check the 983 DMA module to see if the sample is in place, the oven is installed, and the **HEATER** switch is turned on.

C. Press **F1**, the Start key, to begin the experiment.

D. Press **F6**, the Signal Control key, to monitor the experiment.

- Real-time plot can be used to plot raw data in real time.

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Local Keyboard Procedures

Initial Operating Procedures

When using the 983 DMA with the local keyboard/display, install the instrument properly. Before setting up an experiment, make sure you have:

- Made all the necessary cable connections;
- Powered on the unit;
- Installed the available options, if present; and,
- Become familiar with the keyboard.

If all the above steps have been performed, the 983 is ready to be used with the local keyboard/display. The following step-by-step procedures are simple and straight forward. They follow an automatic lead through that is started everytime the **MODULE** key is pressed.

NOTE

The local keyboard is used to set up and control experiments. Data can only be plotted from the 9900 Thermal Analyzer. To transfer data from the 983 DMA to the 9900, press the F2 Module Data File Transfer Key on the File Utilities screen. Refer to Chapter 4 of the 9900 Operator's Manual for further information.

To run an experiment, have all samples and equipment necessary to perform your experiment. Ensure the 983 DMA is properly connected and operational. Mount the sample in the 983 DMA module as directed in the beginning of this chapter.

The Procedures for each mode are given in the following sections:

Local Keyboard Procedures

Resonant Mode

The following information describes a polycarbonate sample being run in the resonant mode from the 983 local keyboard.

For automatic lead through:

Step 1. Setting up the Experimental Parameters

A. Press the **MODULE** key.

B. Use the **INC/DEC** keys and scroll to the DMA resonant mode.

C. Press **ENTER**.

- Experimental parameters for the resonant mode appear in a series of screens.
- Enter the information using the numeric keys. Accept the information using the **ENTER** key.
- The Experimental Parameter screens follow:

Experimental Parameters

D.	Osc Amplitude (mm):	**0.2**
E.	Data Factor:	**4**
F.	Maximum number of runs:	**1**
G.	Change Parameters?	**Yes**

- If the instrument has been recently calibrated, answer **No** and go directly to Step 2.

- When choosing to examine or change the calibration parameters press **Yes**.

H. Reset System Parameters? **No**

- If you answer Yes, all system parameters and constants will be reset to default values.

> **NOTE**
> **The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.**

I.	Length Correction (mm):		**0.00**
	Poisson's Ratio:		**0.44**
J.	Shear Distortion:		**1.50**
	Clamping Distance (mm):		**8.00**
K.	Inertia ($g\text{-}m^2$):		**2.50**
	*Drive Con ($mm/mV\text{-}sec^2$):		**0.0200**
L.	Stiffness (N-m)	@Freq:	**18.00**
	Store: **0.35**	Loss:	**0.2**
M.	Compliance ($\mu m/N$)	@Freq:	**90.00**
	Store: **1.000**	Loss:	**0.020**
N.	Maximum Frequency (Hz):		**120.0**

Threshold Parameters

O.	Threshold	Temp:	**0.05**
	Freq: **0.10**	Drive:	**0.20**

*Drive Con refers to Drive Signal Constant, C'.

Step 2. Method Creation

- Each mode uses the same final steps consisting of Method and Sample ID. The automatic lead-through directly moves to the Method Editor, which can also be attained by pressing the **METHOD** Key.

A. Enter: Method Number: **1**
Name: **5°C/min Ramp**

B. Segment 1 - Equilibrate at **100.00°C**

C. Segment 2 - Ramp **5.00°C/min** to **190°C**

- For more information see Chapter 4.

D. A question appears: Add another segment? **No**

Step 3. Enter Sample Information

- The following information is displayed by the automatic lead through. Sample information can also be attained by pressing the **Sample ID** Key.

- Enter the required sample information.

Sample Information

A. Sample: **Polycarbonate**

B. Sample Size (mm) Length: **25.35**
Width: **12.78** Thickness: **3.12**

C. Operator: **Super Tech**

D. Comment: **Resonance Example**

E. Run Number: **1**

Step 4. Running the Experiment

A. Check to see if the sample is mounted properly, the oven is in place, and the **HEATER** switch is on.

B. Press the START key.

Step 5. Monitoring the Experiment

When choosing to monitor the signals, press the **STATUS** key. Use the **INC/DEC** keys to scroll through the signals. The screen will display the signal values. The top right corner of the screen displays the event state and the store data state. The motor drive state is displayed on the lower line along with the current method segment. As new data is collected, the status display is updated. The signals for resonant mode follow.

Table 3.5
Resonant Mode Status Signals

Run time (min)	Experiment run time
Segment time (min)	Time current segment has been running
Arm position (mm)	Absolute arm position or center of oscillation
Amplitude (mm)	Oscillation amplitude
Drive signal (mV)	Motor drive power (gain)
Offset (mV)	Motor drive offset
Frequency (Hz)	Resonant frequency
Flex store (GPa)	Calculated flex store modulus
Flex loss (GPa)	Calculated flex loss modulus
Shear store (GPa)	Calculated shear store modulus
Shear loss (GPa)	Calculated shear loss modulus
Tan delta	Calculated tan delta
Set point temp (°C)	Heater control set point temperature
Heater power (watts)	Applied heater power

Fixed Frequency Mode

The following information describes a polycarbonate sample being run in the fixed frequency mode from the 983 local keyboard.

For automatic lead through:

Step 1. Setting up the Experimental Parameters

A. Press the **MODULE** key.

B. Use the **INC/DEC** keys and scroll to DMA fixed frequency mode.

C. Press **ENTER**.

- Experimental parameters for the fixed frequency mode appear in a series of screens.
- Enter the information using the numeric keys. Accept the information by using the **ENTER** key.

Experimental Parameters

D. Osc Amplitude (mm): **0.50**

E. Frequency (Hz): **1.000**

- This is the value which will be used in the Fixed Frequency mode to run the experiment.

F. Data Factor: **4**

G. Equilibrate Cycles: **5**

H. Measurement Points: **5**

I. Maximum number of runs: 1

J. Change Parameters? **Yes**

- If the instrument has been recently calibrated, answer **No** and go directly to Step 2.

- When choosing to examine or change the calibration parameters press **Yes**.

K. Reset System Parameters? **No**

- If the answer is Yes, all system parameters and constants will be reset to default values.

NOTE

The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

L.	Length Correction (mm):		**0.00**
	Poisson's Ratio:		**0.44**
M.	Shear Distortion:		**1.50**
	Clamping Distance (mm):		**8.00**
N.	Inertia (g-m^2)		**2.50**
	*Drive Con (mm/mV-sec^2):		**0.0200**
O.	Stiffness (N-m)	@Freq:	**18.00**
	Store: **0.35**	Loss:	**0.2**
P.	Compliance (μm/N)	@Freq:	**90.00**
	Store: **1.000**	Loss:	**0.020**
Q.	Maximum Frequency (Hz):		**120.0**

*Drive Con refers to Drive Signal Constant, C'.

Threshold Parameters

R. Threshold Temp: **0.05**
Phase: **0.10** Drive: **0.20**

Step 2. Method Creation

- Each mode uses the same final steps consisting of Method and Sample ID. The automatic lead through directly moves to the Method Editor, which can also be attained by pressing the **METHOD** Key.

A. Enter: Method Number: **1**
Name: **5°C/min Ramp**

B. Segment 1 - Equilibrate at **100.00°C**

C. Segment 2 - Ramp **5.00°C**/min to **190°C**

- For more information see Chapter 4.

D. A question appears: "Add another segment?" **No.**

Step 3. Entering Sample Information

- The following information is displayed by the automatic lead through. This sample information can be attained by pressing the **Sample ID** key.

- Enter the required sample information.

Sample Information

A. Sample: **Polycarbonate**

B. Sample Size (mm) Length: **25.35**
Width: **12.78** Thickness: **3.12**

C. Operator: **Super Tech**

D. Comment: **Fixed Frequency Example**

E. Run Number: **2**

Step 4. Running the experiment

A. Check to see if the sample is mounted properly, the oven is in place, and **HEATER** switch is on.

B. Press the **START** key.

Step 5. Monitoring the Experiment

When choosing to monitor the signals, press the **STATUS** key. Use the **INC/DEC** keys to scroll through the signals. The keyboard screen will display the signal values. The top right corner of the screen displays the event state and the stored data state. The motor drive state is displayed on the lower line along with the current method segment. As new data is collected, the display is updated. The signals for fixed frequency mode follow.

Table 3.5
Fixed Frequency Mode Status Signals

Run time (min)	Experiment run time
Segment time (min)	Time current segment has been running
Arm position (mm)	Absolute arm position or center of oscillation
Amplitude (mm)	Oscillation amplitude
Drive signal (mV)	Motor drive power (gain)
Offset (mV)	Motor drive offset
Frequency (Hz)	Fixed frequency
Phase (rad)	Fixed Frequency Phase
Flex store (GPa)	Calculated flex store modulus
Flex loss (GPa)	Calculated flex loss modulus
Shear store (GPa)	Calculated shear store modulus
Shear loss (GPa)	Calculated shear loss modulus
Tan delta	Calculated tan delta
Set point temp (°C)	Heater control set point temperature
Heater power (watts)	Applied heater power

Multiple Fixed Frequencies

Experiments with multiple fixed frequencies are run like standard fixed frequency experiments. The only difference is in the method creation where the desired frequencies are entered in Measure at Frequency segments. The procedure below describes the differences in setting up a multiple frequency experiment.

Step 1. Set up the instrument in the fixed frequency mode as described in the previous section.

Step 2. Press the **METHOD** key to create a method.

A. Enter: Method Number: **2**
Name: **Multiple Frequencies**

B. Segment 1 - Equilibrate at **130.00°C**

C. Segment 2 - Measure at Frequency **0.250** Hz

D. Segment 3 - Measure at Frequency **0.500** Hz

E. Segment 4 - Measure at Frequency **1.000** Hz

F. Segment 5 - Measure at Frequency **2.000** Hz

G. Segment 6 - Increment **2.00°C**

H. Segment 7 - Repeat Segment 2 til **160.00°C**

I. A question appears: "Add another segment?" **No**

Step 3. Run the experiment.

A. Press the **START** key.

- The experiment is run like a standard fixed frequency experiment.

Local Keyboard Procedures

Stress Relaxation Mode

The following information describes a polycarbonate sample being run in the stress relaxation mode from the 983 local keyboard.

For automatic lead through:

Step 1. Setting up the Experimental Parameters

A. Press the **MODULE** key.

B. Use the **INC/DEC** keys and scroll to the DMA stress relaxation mode.

C. Press **ENTER.**

- Experimental parameters for the stress relaxation mode appear in a series of screens.
- Enter the information using the numeric keys. Accept the information using the **ENTER** key.
- The Experimental Parameter screens follows.

Experimental Parameters

D.	Displacement (mm):	**0.50**
E.	Data Factor:	**4**
F.	Log Factor:	**1.2**
G.	Equilibrate Criteria: (0=rate; 1=time)	**1**
H.	Equilibrate Time (min):	**60.00**
I.	Maximum number of runs:	**1**

J. Change Parameters? **Yes**

- If the instrument has been recently calibrated, answer **No** and go directly to Step 2.

- When choosing to examine or change the calibration parameters, press **Yes**.

K. Reset System Parameters? **No**

- If answering Yes, all system parameters and constants will be reset to default values.

> **NOTE**
>
> **The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own set of constants.**

L.	Length Correction (mm):		**0.00**
	Poisson's Ratio:		**0.44**
M.	Shear Distortion:		**1.50**
	lamping Distance (mm):		**8.00**
N.	Inertia (g-m^2):		**2.50**
	*Drive Con (mm/mV-sec^2):		**0.0200**
O.	Stiffness (N-m)	@Freq:	**18.00**
	Store: **0.35**	Loss:	**0.2**
P.	Compliance (μm/N)	@Freq:	**90.00**
	Store: **1.000**	Loss:	**0.020**
Q.	Maximum Frequency (Hz):		**120.0**

Threshold Parameters

R.	Threshold	Temp:	**0.05**
	Arm Pos: **0.05**		

* Drive Con refers to the Drive Signal Constant, C'.

Step 2. Method Creation

- Each mode uses the same final steps consisting of Method and Sample ID. The automatic lead through directly moves to the Method Editor, which can also be attained by pressing the **METHOD** Key.

A. Enter: Method Number: **3**
Name: **10°C Inc & Displace**

B. Segment 1 - Equilibrate at **60.00**°C

C. Segment 2 - Displace Time **100.00** Rcvr **0.00**

D. Segment 3 - Increment **10.00**°C

E. Segment 4 - Repeat Segment **2** til **140.00**°C

- For more information see Chapter 4.

F. A question appears: Add another segment? **No**

Step 3. Entering Sample Information

- The following information is displayed by the automatic lead through. Sample information can also be attained by pressing the **Sample ID** key.

- Enter the required sample information.

Sample Information

A. Sample: **Polycarbonate**

B. Sample Size (mm) Length: **25.35**
Width: **12.78** Thickness: **3.12**

C. Operator: **Super Tech**

D. Comment: **Stress Relaxation Example**

E. Run Number: **3**

Step 4. Running the Experiment

A. Check to see if the sample is mounted properly, the oven is in place, and the **HEATER** switch is on.

B. Press the **START** key.

Step 5. Monitoring the Experiment

When choosing to monitor the signals press the **STATUS** key. Use the **INC/DEC** keys to scroll through the signals. The keyboard screen will display the signal values. The top right corner of the screen displays the event state and the stored data state. The motor drive state is displayed on the lower line along with the current method segment. As new data is collected, the status display is updated. The signals for the stress relaxation mode follow.

Table 3.5
Stress Relaxation Mode Status Signals

Run time (min)	Experiment run time
Segment time (min)	Time current segment has been running
Arm position (mm)	Absolute arm position
Amplitude (mm)	Arm position relative to the start of displacement
Drive signal (mV)	Motor drive power (gain)
Offset (mV)	Motor drive offset
Decay time (min)	Stress relaxation decay time
Set point temp (°C)	Heater control set point temperature
Heater power (watts)	Applied heater power

Creep Mode

The following information describes a polycarbonate sample being run in the creep mode from the 983 local keyboard.

For automatic lead through:

Step 1. Setting up the Experimental Parameters

A. Press the **MODULE** key.

B. Use the **INC/DEC** keys and scroll to the DMA creep mode.

C. Press **ENTER**.

- Experimental parameters for the creep mode appear in a series of screens.
- Enter the information using the numeric keys. Accept the information using the **ENTER** key.
- The Experimental Parameter screens are as follow.

Experimental Parameters

D.	Displacement (mm):	**0.50**
E.	Temperature Dependent Load?	**Yes**
F.	Data Factor:	**4**
G.	Log Factor:	**1.2**
H.	Equilibrate Criteria:	**1**
I.	Equilibrate Time (min):	**60.00**

J. Maximum number of runs: **1**

K. Change Parameters? **Yes**

- If the instrument has been recently calibrated, answer **No** and go directly to Step 2.

- To examine or change the calibration parameters, press **Yes.**

L. Reset System Parameters? **No**

- If answering Yes, all system parameters and constants will be reset to default values.

NOTE

The following values are typical values and should not be taken as actual instrument constants. Each unit should be calibrated to determine its own individual set of constants.

M.	Length Correction (mm):		**0.00**
	Poisson's Ratio:		**0.44**
N.	Shear Distortion:		**1.50**
	Clamping Distance (mm):		**8.00**
O.	Inertia (g-m^2):		**2.50**
	*Drive Con (mm/mV-sec^2):		**0.0200**
P.	Stiffness (N-m)	@Freq:	**18.00**
	Store: **0.35**	Loss:	**0.2**
Q.	Compliance (μm/N)	@Freq:	**90.00**
	Store: **1.000**	Loss:	**0.020**
R.	Maximum Frequency:		**120.0** (Hz)

* Drive Con refers to Drive Signal Constant, C'.

Threshold Parameters

R. Threshold Temp: **0.05**
Arm Pos: **0.05**

Step 2. Method Creation

- Each mode uses the same final steps consisting of Method and Sample ID. The automatic lead-through moves directly to the Method Editor which can also be attained by pressing the **METHOD** Key.

A. Enter: Method Number: **3**
Name: **10°C Inc & Display**

B. Segment 1 - Equilibrate at **60.00°C**

C. Segment 2 - Displace Time **100.00** Rcvr **0.00**

D. Segment 3 - Increment **10.00°C**

E. Segment 4 - Repeat 2 til **140.00°C**

- For more information see Chapter 4.

F. A question appears: Add another segment? **No.**

Step 3. Enter Sample Information

- The following information is displayed by the automatic lead-through. Sample information can also be attained by pressing the **Sample ID** key.

- Enter the required sample information.

Sample Information

A. Sample: **Polycarbonate**

B. Sample Size (mm): Length: 25.35
Width: 12.78 Thickness: 3.12

C. Operator: **Super Tech**

D. Comment: **Creep Example**

E. Run Number: **4**

Step 4. Running the Experiment

A. Check to see if the sample is mounted properly, the oven is in place, and the **HEATER** switch is on.

B. Press the **START** key.

Step 5. Monitoring the Experiment

When choosing to display the signals press the **STATUS** key. Use the **INC/DEC** keys to scroll through the signals. The screen will display the signal values. The top right corner of the screen displays the event state and the stored data state. The motor drive state is displayed on the lower line along with the current method segment. As new data is collected, the status display is updated. The signals for creep mode follow.

Table 3.5
Creep Mode Status Signals

Run time (min)	Experiment run time
Segment time (min)	Time current segment is running.
Arm position (mm)	Absolute arm position
Amplitude (mm)	Arm position relative to the start of displacement
Drive signal (mV)	Motor drive power (gain)
Offset (mV)	Motor drive offset
Decay time (min)	Creep decay time
Set temperature (°C)	Heater control set point temperature
Heater power (watts)	Applied heater power

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Subambient Operation

For subambient thermal methods, the 983 DMA uses liquid nitrogen, combined with nitrogen gas, as a coolant. The Liquid Nitrogen Coolant Accessory (LNCA-II) is an optional accessory which can be used with the 983 to provide a source of liquid and gas coolant.

Additional LNCA operation information can be found in the manual that accompanies the accessory, but the following information will be helpful when operating the LNCA with the 983. If you own an LNCA-II, the cooling process will be automatically controlled by way of the 983 DMA. For those who have an LNCA-I or other liquid nitrogen tanks or accessories, the coolant must be controlled manually.

A typical operating procedure would be:

1. Install the locking pins.

2. Mount the sample at room temperature with the clamps finger-tight.

3. Replace the oven, drive assembly, and the access cover on the 983. Plug the oven outlet port with the stopper.

4. Cool the sample down to the starting temperature. This can be done automatically by the 983 DMA with the LNCA-II. Start a method that has as its first two segments: 1) a Motor Drive Off (0) segment, followed by 2) an Initial Temperature segment to the starting temperature. The Motor Drive Off segment prevents the 983 from trying to start the motor when the method is started. The Initial Temperature segment will use the LNCA-II to cool the sample to the starting temperature and then hold the temperature there until the START key is pressed.

5. After the sample has equilibrated at the initial temperature for several minutes, turn the **HEATER** switch OFF and remove the oven.

6. Torque the sample to 10 in.-lb. (1.1N-m)

7. Replace the oven.

8. Open the access cover in the drive assembly and remove the locking pins.

9. Replace the access cover.

10. Turn the **HEATER** switch back ON and let the sample return to the starting temperature.

11. Start the motor. From the 9900, use **F1, the Motor Drive On/Off** key on the Signal Control screen. Or use the **MOTOR ON/OFF** key on the local keyboard.

12. After the motor has started and the sample has equilibrated, start the experiment by pressing the **START** key.

Manual Subambient Operation

The 983 DMA can also be used with a manually controlled cooling source. In this case, the sample can be cooled below the starting point and the clamps torqued before the method is started. Once the method is started, the coolant should be gradually adjusted to keep the DMA heater power between 30 and 40 watts. Avoid rapid changes in the coolant flow because they may cause non-linear heating rates which will be reflected in noisy or discontinuous curves.

The heater power is displayed by the **STATUS** key on the local keyboard and on the **Signal Control** screen on the 9900.

General Guidelines

The following is a list of general guidelines which should prove helpful when using the 983.

1. When operating in resonant mode, adjust the sample size to obtain a resonant frequency between 20 and 40 Hz for the most accurate results.

2. Select a clamp using the chart provided on pages 3-7 and 3-8.

3. When running a low loss sample in the fixed frequency mode, stay below the resonant frequency; otherwise, problems may occur in motor start-up.

4. When running a sample less than 6 mm (the suggested sample length) remove the drip pan, loosen the thermocouple and then the guidebar. Carefully move the guidebar out to the front of the instrument and move the thermocouple outside the sample arms. Slide the guidebar back to position and tighten. Slide the thermocouple in position and tighten. Replace the drip pan.

5. The recommended experimental or module parameters for analyzing a sample in the fixed frequency mode are: Osc Amplitude which should be increased by a factor of 2 or 3 to what is normally used in the resonant mode. See Figure 3.33.

   The recommended number of Equilibrate Cycles is 4 to 5 (unless operating at frequencies between 1 and 10 Hz where the recommended value is 10 to 20).

   If operating at frequencies between 1 and 10 Hz only, the recommended value for the Measurement Points is 10 to 20.

6. It is best to sweep from low to high frequencies in a fixed frequency multiplexing experiment.

7. When running low tan delta samples in the fixed frequency mode, perform the phase angle calibration daily.

> **NOTE**
>
> **When operating in Fixed Frequency and the phase angle calculation is lost, ERR 82 will appear on the screen.**

8. If interested in extending the range of the 983 to frequencies above 10 Hz, operate in the resonant mode.

9. Run an experiment in the stress relaxation mode when trying to determine how material behaves under an applied load at a fixed deformation.

10. Use the creep mode to specifically examine the steady-state flow properties of a material.

11. The characterizations of material at lower frequencies provide the most useful information.

12. When running a sample at ambient temperatures use a set point temperature at 0°C which will prevent the oven from turning on.

13. When running a sample from subambient to elevated temperatures with a nitrogen purge, manually switch from liquid nitrogen cooling to the nitrogen gas purge set between 150 and 200°C. The LNCA II does not supply nitrogen gas above 300°C.

14. See Figure 3.34 to estimate the % of sample strain at different amplitude settings.

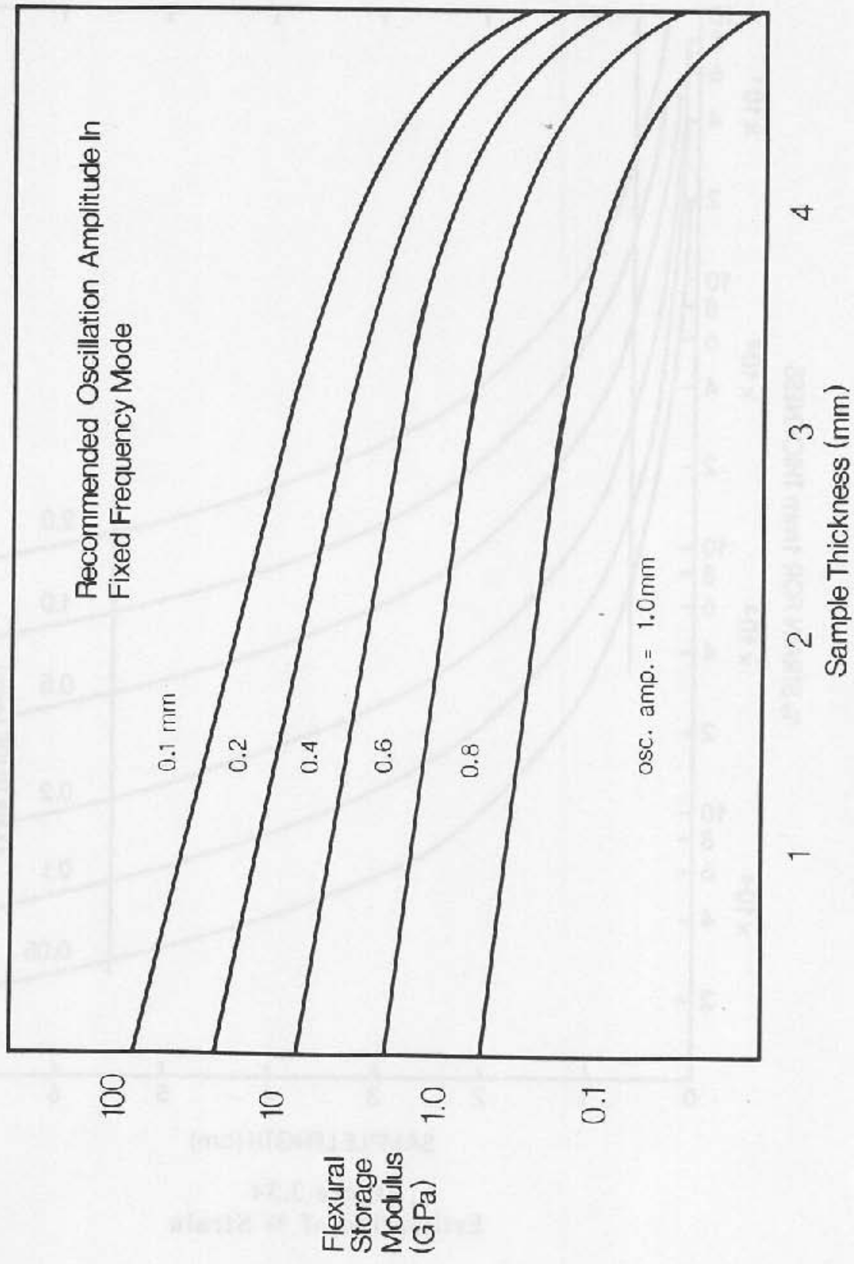


Figure 3.33
Recommended Oscillation Amplitude
in Fixed Frequency Mode

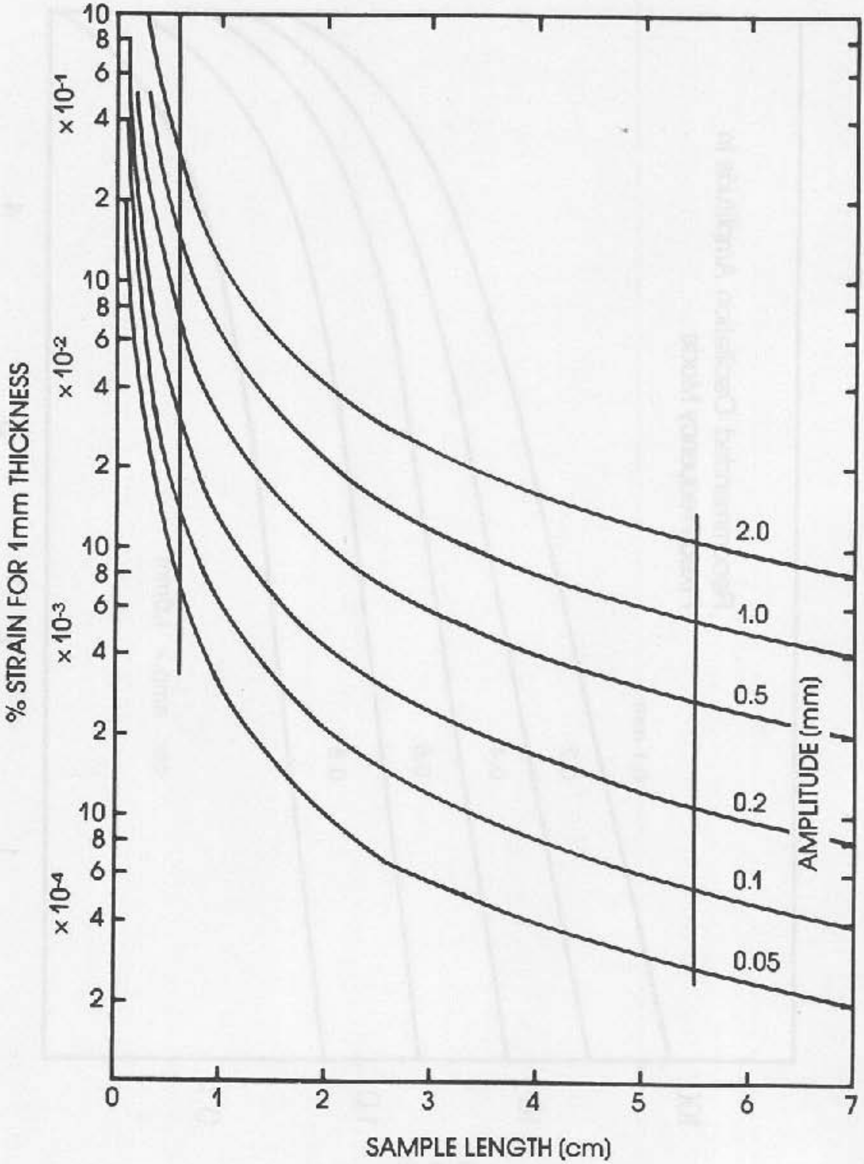


Figure 3.34
Estimation of % Strain

CHAPTER 4

Technical Notes

CHAPTER 4

Technical Notes (continued)

Technical Notes

Principles of the 983 DMA

Dynamic Mechanical Analysis is one of the most sensitive single techniques available for characterizing and interpreting the mechanical behavior of materials. The concept of the DMA is based on observing the viscoelastic response of materials subjected to a small oscillatory strain. The Du Pont 983 DMA uses a flexural bending deformation mode of strain, but also has the capability for other deformations such as shear.

The technique separates the viscoelasticity of a material into the two components of Modulus (E*): a real part, which is the elastic modulus (E'); and an imaginary part, which is the damping or viscous component (E"). The standard complex variable notation is: $E^* = E' + iE''$. This separation of measurements into the two components describing two independent processes within the materials-elasticity (energy storage) and viscosity (energy dissipation) is the fundamental feature of dynamic mechanical analysis that distinguishes it from other mechanical testing techniques.

The need for such capabilities is two-fold:

1. Materials have viscoelastic properties that can be correlated with the end-use parameters.
2. Viscoelastic measurements are sensitive indicators of internal structure, and as such can be used to develop structure/property relationships of materials.

The ability to measure viscoelastic or rheological properties as they vary with temperature makes the DMA technique useful in:

- Predicting end-use product performance
- Correlating impact stability and damping
- Measuring glass transition and secondary transition temperatures
- Determining the compatibility of blends of polymers
- Understanding and optimizing the curing phenomena in thermosets
- Detecting plasticizers and residual solvents in polymers
- Predicting the physical aging of amorphous materials
- Correlating sound or vibration dissipation with damping
- Measuring heat build-up due to repetitive mechanical deformation.

Viscoelasticity

The term "viscoelastic" is used to describe materials whose properties fall between those of a solid (elastic) and those of a liquid (viscous). Polymers as a class of materials often exhibit obvious viscoelastic behavior such as creep and stress relaxation.

The elastic component is best described in terms of a spring in which all energy during deformation is stored and can be regained by releasing the stress. The modulus relationship for such a system is described by the slope of the stress vs. strain plot in Figure 4.1.

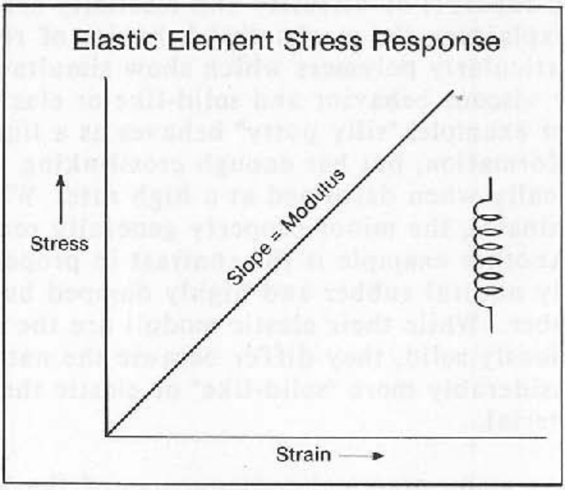


Figure 4.1
Stress vs. Strain Plot

The viscous component is analogous to a dashpot, which resists stretching with a force proportional to the rate of strain. The slope of the stress vs. strain rate curve is defined as viscosity. See Figure 4.2. The deformation energy is dissipated as heat and cannot be recovered.

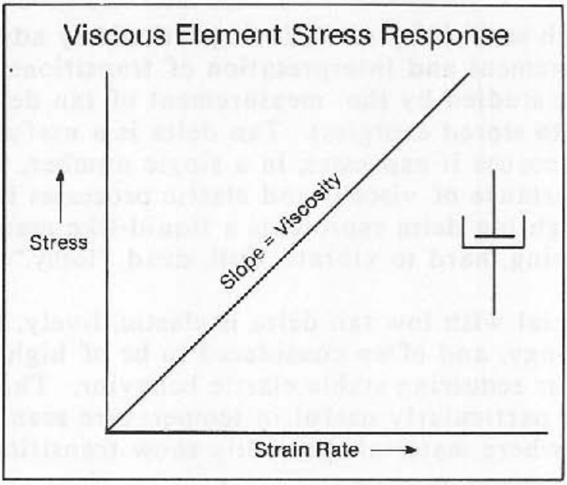


Figure 4.2
Viscosity

The two concepts of viscosity and elasticity are very valuable in explaining the mechanical behavior of real materials, particularly polymers which show simultaneous liquid-like or viscous behavior and solid-like or elastic behavior. For example, "silly putty" behaves as a liquid in long term deformation, but has enough crosslinking to make it behave elastically when deformed at a high rate. When one property dominates, the minor property generally remains important. Another example is the contrast in properties between lively natural rubber and highly damped butyl synthetic rubber. While their elastic moduli are the same and both are obviously solid, they differ because the natural rubber is considerably more "solid-like" or elastic than the synthetic material.

There are many practical consequences of the "dual behavior" of materials. In solids, the liquid-like, energy-absorbing behavior can impart impact resistance and dampen out potentially harmful or annoying structural vibrations. In elastomers, which undergo repeated flexing, viscous energy dissipation causes heat build-up, sometimes with disasterous consequences, as in racing tire failures. The same property, however, also increases cornering traction. Thus, careful control and optimization of viscous damping properties are very important in automobile tires.

The high sensitivity of DMA is particularly advantageous in the measurement and interpretation of transitions that, by tradition, are studied by the measurement of tan delta (the ratio of lost to stored energies). Tan delta is a useful calculation because it expresses, in a single number, the relative importance of viscous and elastic processes in a material. High tan delta represents a liquid-like material, given to flowing, hard to vibrate, dull, dead, "lossy."

A material with low tan delta is elastic, lively, bouncy, resilient, springy, and often considered to be of high quality in applications requiring stable elastic behavior. This tan delta ratio is particularly useful in temperature scanning experiments where materials generally show transitions in their viscoelastic properties. These liquid-like, energy-absorbing processes generally originate from molecular motions which, in polymers, can be classified into three types. These are:

TYPE I motions involve long enough segments of the polymer chain to force other chains to move out of the way. These "cooperative main chain motions" become increasingly prevalent at T_g and can be used to define the glass transition temperature of a material. The glass transition of semicrystallane 66 nylon, for example, is ascribed to the alpha transition at 25 °C as shown in Figure 4.3.

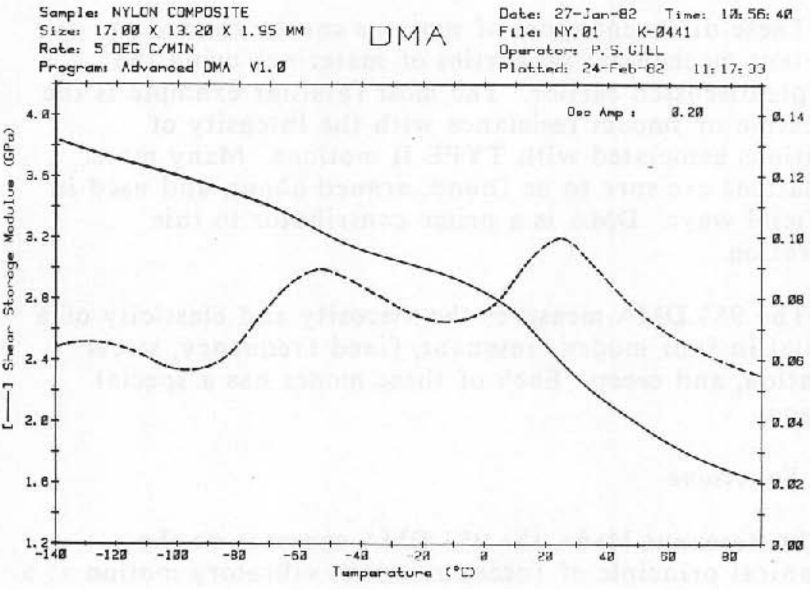


Figure 4.3
Transitions

TYPE II motions involve shorter main chain segments than those involved in alpha transitions. For this reason, they can occur below the T_g of a material. An example of this kind of motion is the shear loss modulus peak at -60 °C shown in Figure 4.3 This has been assigned to the local main chain motions of the amide groups found in the main chain structure of the polyamide networks.

TYPE III motions also involve short chain segments, but these segments are off the main chain axis and do not have as long a range of influence as the first two types; they can be by-passed by any waves of energy in the main chain of the polymer.

These different types of motions can be related to important mechanical properties of materials, using the concepts discussed earlier. The most familiar example is the correlation of impact resistance with the intensity of transitions associated with TYPE II motions. Many more correlations are sure to be found, argued about, and used in beneficial ways. DMA is a prime contributor to this exploration.

The 983 DMA measures the viscosity and elasticity of a material in four modes: resonant, fixed frequency, stress relaxation, and creep. Each of these modes has a special function.

Mode Functions

In **Resonant Mode**, the 983 DMA operates on the mechanical principle of forced resonant vibratory motion at a fixed amplitude (strain), which is selected by the operator. The arms and sample are displaced by the electromagnetic driver, which subjects the sample to a fixed deformation and sets the system in resonant oscillation. The amplitude and frequency of oscillation are measured by the LVDT. That measurement, together with the amount of energy sent to the driver, is used to calculate the desired viscoelastic properties. This mode is particularly useful for analyzing stiff, low-loss samples such as polymers, filled polymers, reinforced plastics, and composites.

When operating in the **Fixed Frequency Mode**, the operator can specify the frequency of oscillation, from 0.001 to 10 Hz. In this mode, a sinusoidal driver signal (applied stress) forces a sample to undergo oscillatory motion at a fixed deformation (strain). Energy dissipation in the sample causes the resulting strain to be out of phase with the driver signal. In other words, because the sample is viscoelastic, maximum strain does not occur at the same instant as maximum stress. This phase shift or lag is defined as phase angle. By knowing the sample geometry and measuring the driver signal and phase angle, viscoelastic properties can be calculated. This mode is particularly useful for analyzing soft high-loss samples such as filled rubbers and high-damping polymers.

In the **Stress Relaxation Mode** of operation, the 983 DMA measures the stress decay at a constant sample deformation as a function of time and temperature. The sample is displaced and held at a constant deformation. The displacement direction is adjusted to keep the sample moving near or through the zero arm position. The amount of driver energy required to maintain that level of strain is then monitored as a function of time. The stress is then removed and the sample is allowed to return to its equilibrium position. The sample recovery is monitored as a function of time. Although the instrument compensates for the pivot restoring forces on the sample, the recovery values for a soft sample will only be qualitative. This process can be repeated at various temperatures to obtain a set of stress relaxation curves over a temperature region.

When using **Creep Mode**, the 983 DMA applies a fixed load or stress to the sample and measures the resulting deformation. The sample is stressed, and the sample deformation is measured as a function of time. The displacement direction can either be optimized to extend the useful range of the measurement or held constant. The sample is then released, and sample recovery is monitored as a function of time. Although the instrument compensates for the pivot restoring forces on the sample, the recovery values for a soft sample will only be qualitative. This process can be repeated at various temperatures to obtain a set of creep curves over a temperature region.

Table 4.1
983 Operating Modes and Data Signals

Mode	Signal A	Signal B	Signal C
Fixed Frequency	Frequency (Fixed)	Driver Force	Phase Angle
Resonant Frequency	Frequency	Driver Force	not used
Stress Relaxation	Arm Displacement (Fixed)	Driver Force	Decay Time
Creep Measurement	Arm Displacement	Driver Force (constant)	Decay Time

Experimental Parameters

The experimental parameters provide the means of entering the experiment information. There are three categories of experimental parameters: 1) Mode Dependent Parameters; 2) Instrument Calibration Parameters; and 3) Sample Parameters.

Mode Dependent Parameters

Mode Parameters are required to execute or control the instrument's operation in a particular mode. The table on the following page lists the parameters which are specific to each run mode.

Table 4.2
Mode Parameters

Resonant Mode Oscillation Amplitude * Data Factor * Threshold * Number of Runs	**Fixed Frequency Mode** Frequency Oscillation Amplitude * Data Factor Equilibrate Cycles Measurement Points * Threshold * Number of Runs
Stress Relaxation Mode Displacement * Data Factor Log Factor Equilibrate Criteria Equilibrate Rate Equilibrate Time * Threshold * Number of Runs	**Creep Mode** Displacement Temperature Dependent Load * Data Factor Log Factor Equilibrate Criteria Equilibrate Rate Equilibrate Time * Threshold * Number of Runs

* Indicates parameters common to all run modes.

Location

9900 Mode Selection:	On the Experimental Parameters screen, press **F4,** the Mode Select key. Enter the mode dependent parameters under **F5, Module Parameters.**
Local Keyboard Mode Selection:	Press the **MODULE** key.

Data Factor - The number of raw data values that are integrated together to produce a data point. The data factor is effective only when a method is running.

Displacement - The initial arm deflection distance used in stress relaxation and creep modes.

Equilibrate Criteria - Selects the Equilibrate Rate or Equilibrate Time in stress relaxation and creep experiments.

Equilibrate Cycles - The number of oscillations the sample is subjected to, at a new frequency in order to reach mechanical equilibrium. Data collection starts after this equilibrium period.

Equilibrate Rate - The maximum rate of sample dimension change relative to the displacement distance allowed before a stress relaxation or creep sample is considered to be in equilibrium before an experiment can begin.

Equilibrate Time - The time period allowed in a stress relaxation or creep experiment for a sample to equilibrate before being flexed.

Frequency - The number of oscillation cycles completed in one second. This is the frequency at which the method is run in the Fixed Frequency Mode.

Log Factor - The time ratio between saved points in stress relaxation and creep measurements which are used to acquire data at a logarithmic rate.

Measurement Points - The number of data points acquired during a Measure at Frequency Segment.

Number of Runs - The maximum number of runs that can be stored in the module's local memory.

Temperature Dependent Load - The creep mode offers a temperature dependent load option for the sample deformation. When this option is selected the displacement direction is adjusted each time to keep the sample moving near or through the zero arm position and the stress level is adjusted at the beginning of each step to maintain a constant initial displacement. If this option is not selected, the stress and displacement direction from the first step is used for all the subsequent steps.

Threshold - The signal deviation limits (signal-to-noise ratio) used to control the data compression function.

Instrument Calibration Parameters

These parameters are used in the quantitative calculation routines to compensate for the instrument's contribution in the measurements. These parameters are discussed later in this chapter under Calibration Procedures.

Table 4.3
Instrument Calibration Parameters

Length Correction
Clamping Distance
Poisson's Ratio
Shear Distortion
Moment of Inertia
Drive Signal Constant C'
Parallel Stiffness
Series Compliance

Location

9900 Calibration Parameters:	On the Experimental Parameters screen, press **F5**, Module Parameters.
Local Keyboard Calibration Parameters:	Press the **MODULE** key and answer **Yes** to the question, "Change Parameters?"

Instrument Calibration Definitions

Clamping Distance - The distance from the axis of the arm to the inside of the clamp face. Used in modulus calculations, the distance between arm centers is the sample length plus twice the clamping distance.

Drive Signal Constant C' - A conversion factor from drive signal to acceleration.

Length Correction - An empirical correction added to the sample length to account for sample motion that penetrates beyond the clamp faces.

Moment of Inertia - A measure of the effectiveness of mass in motion.

Parallel Stiffness - Instrument calibration constants used to correct the instrument contributions to the modulus of an extremely soft or thin sample.

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.

Series Compliance - The correction terms used for instrument contributions to the modulus of an extremely stiff sample. These terms cover the bending of arms and the distortion of flexure pivots under extreme loads.

Shear Distortion - The distortion in a plane when a substance is under shear deformation.

Sample Parameters

Sample parameters are descriptive parameters which aid in the identification of the experiment.

Table 4.4
Sample Parameters

Sample ID
Sample Size
Operator
Comment
Run Number
* File name

* Denotes the parameter found only on the 9900.

Location

9900 Sample Parameters:	Press **F1**, Sample Information on the Experimental Parameters screen.
Local Keyboard Sample Parameters:	Press the **Sample ID** key.

Sample Parameter Definitions

Comment - Usually a description of the sample or a statement about the experiment.

File Name - Name of the 9900 disk file in which the data will be stored.

Operator - Name of the person initiating the experiment.

Run Number - A multi-digit identification number associated with the data. The run number is automatically incremented at the end of each run.

Sample ID - A sample identification string used to describe the sample, which appears on the reports and plots.

Sample Size - The length, width, and thickness measurements of the sample in relation to its placement in the 983. Length is the sample length between clamp faces. Thickness is the sample dimension parallel to the arms. Width is the sample dimension in the vertical direction. The sample size is necessary for the modulus calculations. For a cylindrical sample, enter the sample radius as either the width or thickness and make the other value zero. The modulus calculations will automatically use the correct geometry equations.

Method Description

A method is a sequence of segments (steps) used to perform an experiment. The segments:

1. Control temperature.

2. Perform specialized procedures (i.e., Measure at Frequency).

3. Control other functions, such as Data Storage.

The method includes an identifying label for an experiment. There are a total of 15 methods available and 60 segments which can be distributed through any or all of the methods. Fourteen segment types are available on the 983. They are listed in Table 4.5. The glossary contains a brief definition for each segment.

Table 4.5
Method Segments

1. Jump to Temperature
2. Equilibrate at Temperature
3. Initial Temperature
4. Ramp
5. Isothermal Time
6. Increment Temperature
7. Measure at Frequency (fixed frequency mode only)
8. Displace/Recover (creep/stress relaxation modes only)
9. Motor Drive
10. Repeat
11. Repeat til Temperature
12. Data Factor
13. Event
14. Data Storage

Some segments are designed to aid the 983 DMA in performing specialized experiments. There are four specialized segments: 1) Increment Temperature segment; 2) Measure at Frequency segment; 3) Displace/Recover segment; and, 4) Motor Drive segment.

Increment Temperature Segment

The Increment Temperature segment raises or lowers the temperature in a step, allows the oven temperature to equilibrate, before beginning the next segment. This segment can be (+) or (-) depending on the direction. This segment is used in multiple frequency, stress relaxation, and creep experiments. For an example, see Chapter 3 regarding multiple fixed frequency experiments being run using the 9900 and the Local keyboard.

Measure at Frequency Segment

The Measure at Frequency segment is used for multiple frequency experiments. This segment is used to set the frequency and acquire data points. The temperature is held constant during this segment. Data storage is automatically turned **On** to collect the data and is turned **Off** at the end of the segment.

Displace/Recover Segment

The Displace/Recover segment is an isothermal segment that:

1. Allows the sample to equilibrate.

2. Displaces and measures the stress relaxation or creep of a sample, depending on the mode, over a specific period of time.

3. Releases the sample and measures the sample recovery over a specified period of time.

Motor Drive Segment

The Motor Drive Segment is used to automatically turn the motor on and off during a method. If the first segment in a method is a Motor Drive off, then the motor will not be started at the beginning of an experiment. Otherwise, the motor will automatically start when the method begins. The Measure at Frequency segment and Displace/Recover segment automatically start the motor.

Method Creation and Editing

Methods contain one or more segments arranged in order of execution. To create or edit a method, select a method number and a name. This name or identification string is normally a description of what the method does and is included in the run data for the experiment. Once a name is given to the method, create the method by selecting the segments appropriate to perform the experiment. This includes completing the parameter information.

When editing a currently running method, the parameters can be changed, but the segments cannot be changed, added, or deleted. This is especially useful in modifying segments that normally cannot be modified while they are being executed such as the Repeat til Temperature segment.

The following table lists the method editing functions and the location of the procedures on the 9900 and local keyboards.

Table 4.6
Function Key Descriptions and Procedures

Function	Procedure
Change segment Types	9900: Select **F1, Change Segment Type** key. Local: Use the **type** entry on the top line of the display.
Insert a segment in the middle of a method	9900: Select **F2, Insert Segment** key. Local: Use **EDIT** function 1.
Delete a segment	9900: Select **F3, Delete Segment** key. Local: Use **EDIT** function 2.
Restore a deleted segment	9900: Select **F4, Restore Segment.** Local: Not available.
Copy a method	9900: Select **F2** on the main method edit screen. Local: Use **EDIT** function 4.
Delete a method	9900: Select **F3** on the main method screen. Local: Use **EDIT** function 3.
Print a method	9900: Select **F4, Print Method.** Local: Not available.

Table 4.6
Function Key Descriptions and Procedures (Continued)

Function	Procedure
Selecting a method to run	9900: Use **F2 SELECT METHOD** key, on the Experimental Parameters screen. Local: Use the **METHOD** key.

Starting an Experiment

Beginning a 983 DMA experiment requires three steps.

1. Mounting the sample correctly.
2. Starting the motor.
3. Starting the method.

Motor Start Up

The motor start up sequence is mode dependent. This sequence automatically checks the flexure of the sample, the measured stiffness, and the locking pin conditions at motor start up.

The resonant mode start up procedure begins with the arm position at zero. The motor then kicks the arm to induce a free decaying oscillation. The decay rate of this oscillation is used to set up the initial drive level for the oscillation.

For the fixed frequency, stress relaxation, and creep modes, the motor measures the sample stiffness, then starts running. In the fixed frequency mode, the motor starts running at the currently selected frequency; in stress relaxation and creep modes, it starts in the equilibrate (undriven) state.

In both resonant and fixed frequency modes, arm oscillation is necessary for the experiment to begin, unless the first segment is a Motor Drive off segment. If the motor does not start in four attempts, (the initial attempt and three retries), something is wrong and an ERR 83 or 84 will occur. If oscillation is lost during the experiment, the method will continue to run. The motor will try to restart until the method is complete or it is manually stopped. During this time, data will not be collected because of its instability.

In multiple fixed frequency experiments, the motor will sometimes lose oscillation when jumping between high and low frequencies, especially if one frequency is above the sample resonance. When this happens, the motor will try to restart at the new frequency. If the 983 DMA cannot start running the new frequency in three attempts, it will skip that frequency and go on to the next segment rather than hold up the experiment. When this occurs, there will be a gap in the data.

The motor itself can be turned on manually by pressing **F1, Motor Drive** on the 9900 Signal Control screen or pressing the **Motor On/Off** key on the 983 local keyboard. This particular key toggles the state of the motor. If the motor is on, the key turns the motor off. If the motor is off, the key turns the motor on. The **STAND-BY** and **REJECT** keys always turn the motor off.

Method Start Up

At method start up, the instrument ensures that it is ready to run an experiment in the selected mode by checking for:

1. The correct mode;
2. Legal segments;
3. An activated **HEATER** switch;
4. A working fuse; and,
5. A correct oven position.

The 983 DMA checks the heater switch, fuse and oven position by applying and measuring the power that actually goes through the oven. If power can not be applied to the oven ERR 93 will appear. The run will start even though the 983 can not control the sample temperature. If other errors appear, such as ERR 60, check the method. Once guaranteed of a working method, the 983 starts executing the method.

To start a method, press the **START** key. To stop a method, press either the **STANDBY** or **REJECT** key.

When a method is running, modify the current method segment by using the Modify Segment function. This function also allows you to terminate a segment and go on to the next segment.

Data Storage

Data storage can only occur while a method is running and is controlled by the following factors:

1. The data factor;
2. The basic data acquisition rate; and,
3. Certain sequences which automatically turn data storage on and off as they are executed.

Data Storage can be turned on and off with the **DATA** key and the Data segment. The Measure of Frequency and Displace/Recover segments turn data storage on and off when they are executed. In the Resonant and Fixed Frequency Modes, data storage is automatically turned on by the first Isothermal or Ramp segment in the method.

Data Compression

The 983 DMA can generate extraordinarily large data files (1 Mbyte or more) during long experiments. To keep the data file size manageable, the 983 uses a data compression scheme to reduce the amount of redundant data that is stored during flat sections of the curve and yet retain high resolution during peaks and transitions. This compression algorithm is active during all data storage periods except the Measure at Frequency and Displace/Recover method segments.

Data compression works by fitting the data to a straight line from the first point. This line is projected to the next data point, and a tolerance window based on the threshold is created around the projected value. If the new data point lies outside that window, the old line segment is terminated and its end point is saved in the data file. A new line is started using the end of the old line and the new data point as the first points in the line. The data window is calculated for the next data point, and the process is repeated. All the data values are saved and the compression is restarted whenever **any** one of the signals exceeds its window. The data is also saved after one hundred points, regardless of the threshold values.

The data compression windows are controlled by the threshold values that are entered under Module Parameters. The temperature threshold is entered in degrees; all other signal thresholds are entered as percentages. This allows the threshold windows to track the signal with a constant signal-to-noise threshold as the sample goes through a transition. A threshold of 0.0 will bypass the compression and save every data point.

The advantage of this compression technique is that regions of constant slope can be represented by a few widely spaced points, while transition regions where the curve's slope is constantly changing will have many data points to precisely define the transition.

Measure at Frequency Segment

The Measure at Frequency method segment, used in multiple fixed frequency experiments, always turns data storage on at the start of the segment; stores the number of data points specified by the "Measurement Points" entry on the Module Parameters page; and then turns data storage off at the end of the measurement. Data compression is **not** used during a Measure at Frequency segment.

Displace/Recover Segment

The Displace/Recover method segment, used to make Stress Relaxation and Creep measurements, turns data storage on at the start of the displacement; stores the data on an exponentially increasing time interval; then turns data storage off at the end of the recovery. This time interval is controlled by the "Log Factor" entry on the Module Parameters page. Data compression is **not** used during a Displace/Recover segment.

Experimental Control Keys

The 983 DMA is designed to work with the 9900 and the local keyboard. The experimental control keys control special functions, such as the execution of thermal programs. These keys start, hold, continue, stop and reject an experiment: turn the motor on and off; alter a program in progress; and control data storage and external event states.

The following is a brief description of each experimental control key and its location on both the 9900 and the local keyboard.

STANDBY

The **STANDBY** key stops the current thermal experiment, discontinues heater power, and stops the motor. Data storage is halted and stored data is retained.

Location

On the 9900:	**F2** is the StandBy key on the Instrument Control screen.
On the Local Keyboard:	The 1st Operate key found on the upper-right corner beside the **REJECT** key.

REJECT

The **REJECT** key cancels the thermal scan in progress and erases the run data. "Reject" is displayed in the status window on the local keyboard after the key is pressed, and the run number of the rejected run is assigned to the next run. This key also stops the motor.

Location

On the 9900:	**F3** on the Instrument Control screen.
On the Local Keyboard:	The first upper-right key.

START and HOLD

The **START** key initiates the thermal program after checking the method. It continues a method from the "Ready" state of an Initial Temperature segment. Once the experiment begins, the **HOLD** key suspends the method, maintaining the current thermal conditions until the key is pressed again. Data collection and run time are not affected by the **HOLD** key. Press **START** to resume. **HOLD** will not function if the method is in the "set-up" state.

Location

On the 9900:	**F1** on the Instrument Control screen.
On the Local Keyboard:	The second key in the right-most column of the keyboard.

MOTOR ON/OFF

The **MOTOR ON/OFF** key turns the motor on and off. The motor is also controlled by the Displace/Recover and Measure at Frequency segments in the method.

Location

On the 9900:	**F1** on the Signal Control screen.
On the Local Keyboard:	The second key down in the right-most column of the keyboard.

MODIFY SEGMENT

The **MODIFY SEGMENT** key provides a "Go to next segment" feature or enables you to alter the segment in progress. The changes are not stored in the method so the program operates as originally created in subsequent runs.

Location

On the 9900:	F3 on the Experimental Parameters screen.
On the Local Keyboard:	The 3rd key down in the second from the right column of keys.

DATA

The **DATA** key provides manual control over data storage. Normally, data collection starts when the program enters the first run state (Ramp or Isothermal) or when the first Data 1 segment occurs in a program. The **DATA** key may be used to start collection sooner, stop collection in progress, or resume collection. Collection is halted by a Data 0 segment.

Location

On the 9900:	**F8** on the Experimental Parameters screen.
On the Local Keyboard:	The 3rd key in the right-most key column.

> **NOTE**
>
> **To obtain the current data and event states, press the STATUS key on the local keyboard. On the 9900, the status will be displayed automatically on the top of the screen.**

GAS

The **GAS** key provides no function with the 983 DMA.

EVENT

The **EVENT** key provides manual control over the external event contact closure located on the back of the instrument. This contact can also be controlled by the Event segment in the method.

Location

On the 9900:	**F7** on the Experimental Parameters screen.
On the Local Keyboard:	The last key in the lower-right corner of the keyboard.

983 and 9900 Status Codes

Status codes are continuously displayed at the top of the 983 and 9900 screens. To view the extended status of an experiment on the 983, press the **STATUS** key. Although the status messages are brief, these messages provide information while monitoring the progress of an experiment.

Table 4.7 Status Codes

Code	Interpretation
A/Z Lim	The automatic zero circuitry which keeps the oscillation centered on zero has reached the limit of its range. The motor will continue to run, but data accuracy is compromised.
Calib	The method is not running; the motor is running in a calibration mode, performing calibration functions.
Cold	The module heater can not supply heat fast enough to keep up with the thermal program. This may be caused by a large ballistic jump, a faulty heater, or a faulty control thermocouple signal.
Complete	The thermal method has finished.
Cooling	The heater is cooling as a result of a Ramp segment.
Creep	The 983 is executing a creep measurement.
Equilib	The temperature is equilibrating to the desired set point.

Table 4.7 Status Codes (continued)

Code	Interpretation
Err xx	An error has occurred (xx will appear as a two-digit code). Press the **HELP** key on the local keyboard or refer to Appendix A for further assistance.
Fixed	The motor is running in the fixed frequency mode, and the method is idle.
Gain Err	The motor gain circuitry has hit a limit of either zero or maximum volts.
Heating	The heater temperature is increasing in response to a ramp segment.
Holding	Thermal experiment conditions are holding; the program is suspended. Press **START** to continue the run.
Hot	The module is at a higher temperature than the set point and cannot remove heat fast enough to follow the thermal program. This is usually caused by a large ballistic jump to a lower temperature or by running a cooling ramp without the LNCA-II.
Initial	The temperature is equilibrating to the desired set point, then "Ready" will be displayed.
Iso	The thermal program is holding the current temperature isothermally.
Jumping	The heater is jumping ballistically to the set point temperature.
Measure	The thermal program is currently executing a Measure at Frequency segment.
Mtr on	The motor is turning on.

Table 4.7 Status Code (continued)

Code	Interpretation
No Osc	The motor lost oscillation. If a method is running, the motor continually tries to restart. If the method is not running, the motor tries to restart three times.
No Pwr	There is no power to the heater. Check the heater switch and fuse.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Press the **START** key.
Recover	The method is executing the recovery portion of a stress relaxation or creep experiment.
Reject	The experiment has been terminated and the data erased.
Res	The motor is running in the resonant mode, but the method is not running.
Set Up	The system is starting the motor prior to starting the first segment of the method.
Stress	The 983 is running in the stress relaxation portion of a Displace Recover segment.
Temp °C	The heater is in STANDBY mode, and the experiment has been terminated.
Temp *	Two-point temperature calibration is active. The heater is in STANDBY mode, the experiment has been terminated, and the motor is not running.

The status codes appear:

1. At the top of the 9900 screen.
2. At the top right of the local keyboard screen.

Method Status

The method status is a 26-character string that describes the current method segment. On the 9900 system the method status for the current module is displayed on the Instrument Control screen. In single and two module systems, the method status is also displayed at the top of the screen. On the local keyboard you can reach method status by pushing the **STATUS** key.

Motor Status

The motor status is visible on the top left hand column of the Signal Control screen on the 9900. This status will appear on the local keyboard when the **STATUS** key is pressed. Motor status information appears on the bottom line.

The motor status is divided into seven states.

1. Res - Running in a resonant mode.
2. Fixed - Running in a fixed frequency mode.
3. Stress - Running in a stress mode.
4. Creep - Running in a creep mode.
5. Idle - Undriven.
6. Calib - Running in a calibration mode.
7. Start - Motor is starting.

Table 4.8
Status Signal Values

Run Time:	min	Experiment run time
Segment Time:	min	Time the current segment has been running
Arm Position:	mm	Absolute arm position or center oscillation
Amplitude:	mm	Current arm position or oscillation amplitude
Drive Signal:	mV	Motor drive power (gain)
Offset:	mV	Motor drive offset
Frequency:	Hz	Oscillation frequency
Phase:	rad	Phase angle
Flex Store:	GPa	Calculated flex store modulus
Flex Loss:	GPa	Calculated flex loss modulus
Shear Store:	GPa	Calculated shear store modulus
Shear Loss:	GPa	Calculated shear loss modulus
Tan Delta:		Calculated Tan Delta
Decay Time:	min	Stress Relaxation and Creep decay time

Table 4.8 (continued)

Set Point Temp:	°C	Heater control set point temperature
Heater Power:	Watts	Applied heater power
GPIB Remote State		9900 keyboard is controlling the 983 (see **LOCAL** key).
GPIB Local Lockout		All 983 keyboard functions, except HELP, are inhibited by the 9900.

LOCAL

The **LOCAL** key functions only on systems with the GPIB interface. When the 983 is in the remote state, only the **HELP** and Operate keys function. Press the **LOCAL** key to transfer control from the remote device, such as the 9900 Thermal Analysis system, to the local keyboard. In the local lockout state, only the **HELP** key functions.

Calibration Procedures

For quantitative analysis of viscoelasticity of samples, the following instrument parameters must be calibrated:

- Moment of inertia of the arms, J
- Pivot spring constant (or parallel storage stiffness), K'
- Drive signal constant, C'
- Parallel loss stiffness, K_n"
- Series storage compliance, J_C'
- Series loss compliance, J_{Cm}"
- Phase zero
- Length correction, ΔL

Two additional parameters used in the calculations are shear distortion and Poisson's ratio. These factors are explained on page 4-54.

Instrument calibration can be performed through the DMA Calibration program or with the 983 local keyboard. The DMA Calibration program was designed to calculate the instrument constants and sample parameters for the DMA. The use of this program is explained in the *DMA Calibration Program Operator's Manual.* The following sections describe the 983 local keyboard calibration procedure.

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.

Frequency of Calibration

With the exception of moment of inertia, phase zero, and length correction, all calibration procedures should be performed once a month. The moment of inertia calibration needs to be performed only when the instrument is first installed or the arms are replaced. If you are running fixed frequency, using low-loss samples, perform the phase zero calibration at the beginning of each working day; once a week is sufficient for high-loss samples. The length correction procedure should be performed each time you use a different sample material or clamping configuration.

Order of Calibration

In most of the calibration routines, one or more parameters or experimental values are retrieved from the previous routine. It is recommended that you follow the calibration procedures as they are presented in this manual. If only a specific parameter requires recalibration, use Table 4.9 to determine which constants must be calibrated first.

Table 4.9
Interdependence of 983 Calibration Procedures

Procedure	Uses Calculations from:
Inertial Moment	--
Spring Constant	Inertial Moment
Drive Signal Constant	Inertial Moment
Series Compliance	Inertial Moment Spring Constant Drive Signal Constant
Phase Zero	Inertial Moment Spring Constant Drive Signal Constant Series Compliance
Length Correction	Inertial Moment Spring Constant Drive Signal Constant Series Compliance

Typical Calibration Values

As you perform the calibrations described in the following sections, check your results to see if they fall within the typical ranges shown in Table 4.10. If any of the constants are significantly outside of this range, contact your service representative.

Table 4.10
Typical Values for 983 Calibration Constants

J	Moment of Inertia	2.4 to 2.7 g m^2
K'	Parallel Storage Stiffness (Spring Constant)	0.30 to 0.40 N m
K_n"	Parallel Loss Stiffness (at frequencies between 15 and 20 Hz)	0.05 to 0.25 N m
C'	Drive Signal Constant	0.015 to 0.026 mm/(mV sec^2)
J_C'	Series Storage Compliance	0.50 to 1.50 μm/N
J_{Cm}"	Series Loss Compliance (at a frequency below 98 Hz)	0.005 to 0.050 μm/N
	Phase angle Standard deviation	<0.0010 rad
ΔL	Length Correction	0 to 5.0 mm

Calibrating with the Local Keyboard

Follow the general guidelines below to calibrate the 983 DMA with the local keyboard. The correct samples to use and procedures for setting up the instrument for each calibration are explained on the 983 screen.

- Review the instructions in Chapter 3 on mounting samples before you begin.
- Start each calibration by pressing the **INST CALIB** key. The name of a calibration procedure will appear on the screen.
- Press the **INC/DEC** keys to scroll through the menu until you see the procedure you want to run. Press **ENTER** to select the procedure.
- Follow the instructions on the screen. As you complete each instruction, use the **ENTER** or **DOWN ARROW** key to go on to the next step. The **UP ARROW** key scrolls backward toward the first instruction.
- Press the **HELP** key for further instructions whenever you have a question.
- Refer to Chapter 3 for instructions on zeroing the arms.
- When the screen requests numeric input, type the value with the numeric keys, then press **ENTER**.
- When you are prompted to accept a frequency or drive signal readout, wait for the readout to stabilize, then press **ENTER**.
- To cancel a measurement, press the **STANDBY** key.
- At the end of each calibration procedure, press **ENTER** to save your results. The results will be stored in the 983 DMA's battery backed-up memory.

- See pages 4-40 to 4-53 for a description of the principles of each calibration. In these descriptions, the word *system* refers to the instrument plus the mounted sample.

Moment of Inertia

Definition

The moment of inertia (J) is a measurement of how the instrument arms resist change in motion, or the amount of energy that can be stored in the moving mass. It is determined primarily by the mass of the arms.

How it is Measured

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:

$$\text{Moment of Inertia} = \frac{\text{Sample Stiffness}}{\text{Resonant Frequency Squared}}$$

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 front end 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:

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

where:

M	= mass of hanging weight (grams)
G	= gravitational acceleration = 9.80 m/sec^2
R	= distance from center of flexure pivot to the clamp face (0.12785 m)
Δp	= change in measured arm position due to mass (m)
	= $p_m - p_0$
f	= resonant frequency
p_m	= arm position with mass
p_0	= arm position without mass

Typical values for J range from 2.4 to 2.7 g m^2.

Pivot Spring Constant

Definition

The pivot spring constant (K', also called parallel storage stiffness) is a measure of the flexibility (springiness) of the pivots. K' 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 4-59).

How it is Measured

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 of the arms 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)

Typical values for K' range between 0.30 and 0.40 N m.

Drive Signal Constant

Definition

The drive signal constant (C') converts measured drive level to motor torque to determine how much energy is being applied to the system. The parallel loss stiffness (K_n"), also measured in this procedure, 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.

How it is Measured

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 up, 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):

$$\frac{A_0 \text{ (arm displacement)}}{V_0 \text{ (drive signal)}}$$

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

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

Typical values for C' range between 0.015 to 0.026 mm/(mV sec^2).

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 used to calculate K_n'':

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

where:

C' = drive signal constant (mm/(mV sec^2))
V_n = drive signal required to maintain oscillation amplitude a_n (mV)
a_n = measured oscillation amplitude (mm)
J = moment of inertia (kg m^2)

K_n'' typically falls between 0.05 and 0.25 N m.

Series Compliance

Definition

The series compliance constants (J_C' and J_{Cm}'') are used to correct for the nonrigid responses of the instrument that occur with very stiff samples.

How it is Measured

In principle, measuring instrument compliance terms requires an infinitely stiff, no-loss sample, which of course does not exist. Thus, 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':

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

where:

J = moment of inertia (kg m²)
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)

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

L = sample length (mm)
W = sample width (mm)
T = sample thickness (mm)
E = flexural modulus of the compliance sample (200 GPa)
B = distance between arm centers (mm)
= sample length plus 2• (clamping distance)
f_m = measured resonant frequency (Hz)

Typical values for J_C' range between 0.50 and 1.50 μm/N.

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

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

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 = 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)

J_{Cm}'' typically falls between 0.005 and 0.050 μm/N.

Phase Zero

Definition

In fixed frequency experiments, the 983 DMA measures the phase lag between the drive signal and the arm position signal. The phase zero calibration corrects for the contribution of the DMA 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.

How it is Measured

In the phase zero calibration, the instrument 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 oscillation frequencies, then fits the measurements to a theoretical curve and calculates the standard deviation of that fit. The standard deviation of the fit should be 0.001 radian or less. All phase correction data is stored internally in the module.

Length Correction

Definition

This corrects for end effects in the jaw clamps, and depends on both sample hardness and the torque applied to the clamp screws. Each sample type and clamping configuration has its own length correction factor.

The value is added to the sample length to correct for sample flexure that extends beyond the clamp face into the center of the clamps. The length correction is most important for shorter samples (<20 mm). Length corrections are normally positive when the correct clamping pressure is used (10 in-lb for stiff samples, 3-5 in-lb for soft samples). In performing a length correction, always start at the longest sample length and work down.

How it is Measured

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 modulus values are then plotted versus ξ . For rectangular samples, ξ is computed as follows:

$$\xi = \frac{1}{L} \left[\frac{2\,(1+\sigma)\,T^2\,\alpha + 3L^2}{2\,(1+\sigma)\,T^2\,\alpha + L^2} \right]$$

For cylindrical samples:

$$\xi = \frac{1}{L}\left[\frac{6\,(1+\sigma)\,r^2\ \alpha + 3L^2}{6\,(1+\sigma)\,r^2\ \alpha + L^2}\right]$$

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 ξ = zero) is corrected storage modulus (E'). The slope of the line is -E' (corrected) ΔL.

Discussion

Typical values 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 in. lb should be used for hard samples (below Tg). If you are running elastomeric (soft) sample experiments, use the constant tension springs on the clamps to maintain a reliable clamping pressure.

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

- Sample stiffness
- Thickness

As the sample stiffness and thickness increase, the length correction will also increase. Figure 4.4 indicates the estimation of the length correction for a given type of sample. You can use the graphs to: estimate ΔL when time does not permit you to carry out an actual length correction measurement; or confirm that the length correction obtained during a measurement is reasonable.

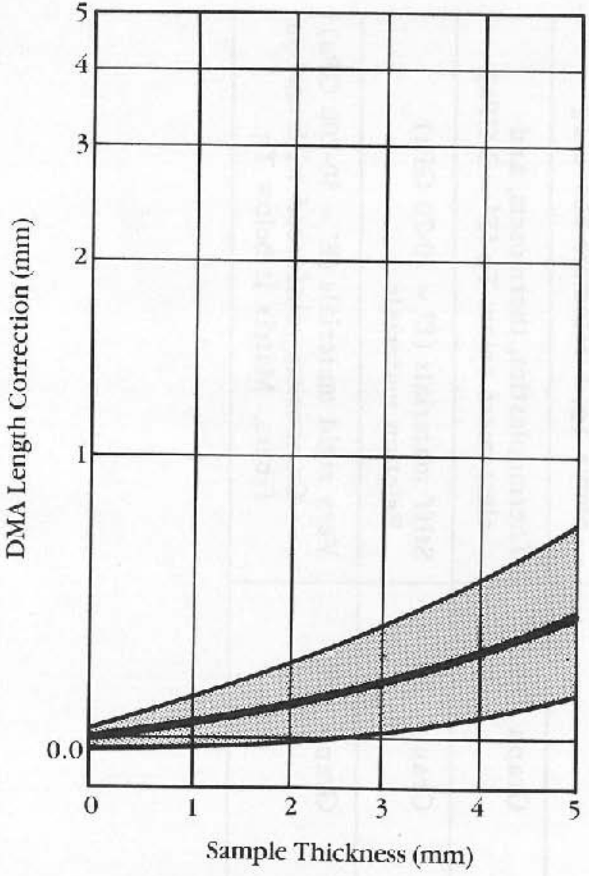
To use the graphs, select the graph for the sample material type using Table 4.11. Then read the estimated length correction for the sample thickness from the graph.

For example, a 3 mm thick sample of polycarbonate at 25°C is to be analyzed. This material is a thermoplastic below its glass transition and has a storage modulus E' of 2.3 GPa. Its classification is therefore II, and a 3 mm thick sample of this type has an estimated ΔL of 1.0 mm.

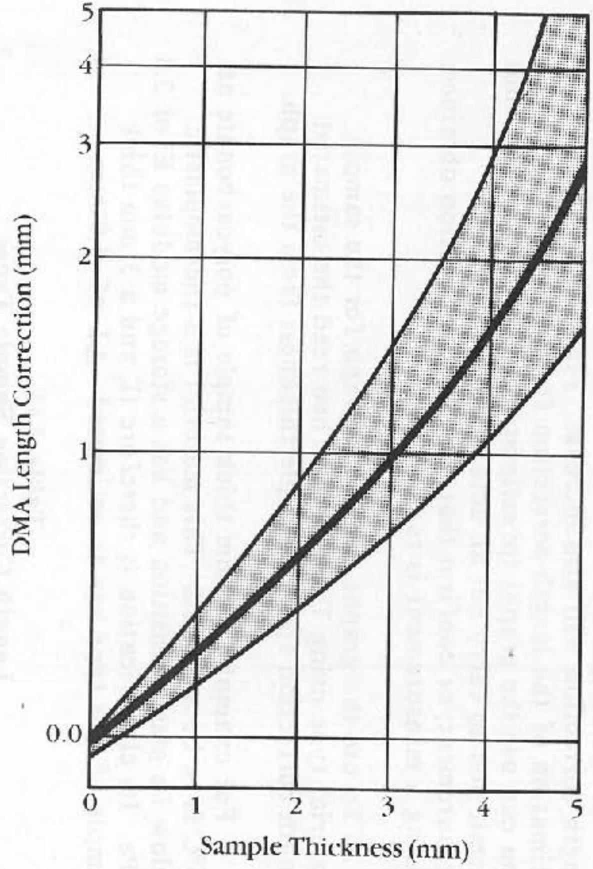
Table 4.11
Length Correction Sample Types

Classification	Type of Sample
Graph I	Soft materials (E'< 0.5 GPa) Thermoplastics/thermosets above Tg; Elastomers above Tg
Graph II	Thermoplastics, thermosets, and elastomers below Tg (E' = 2 GPa)
Graph III	Stiff materials (E' = 10-20 GPa) Friction materials
Graph IV	Very rigid materials (E' = 50-200 GPa) Composites reinforced with carbon fibers. Matrix is below Tg.

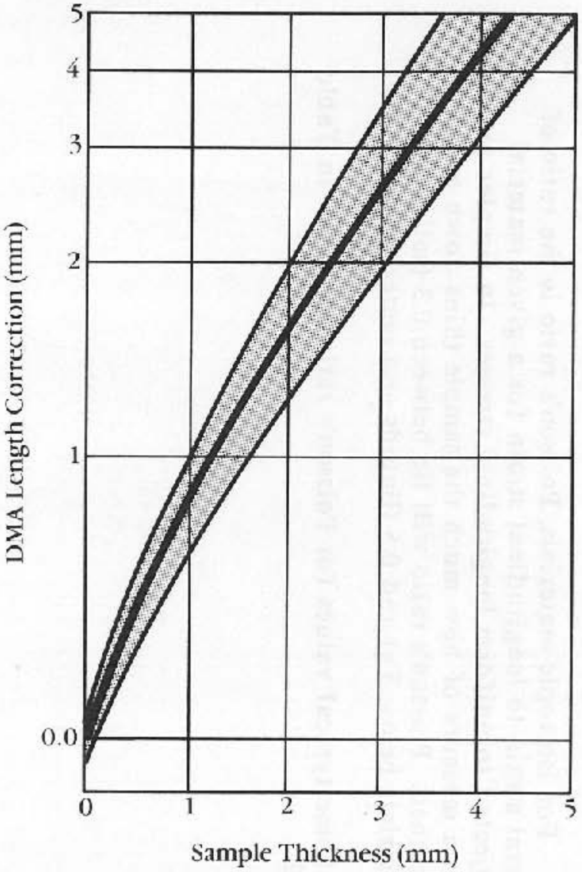
Figure 4.4



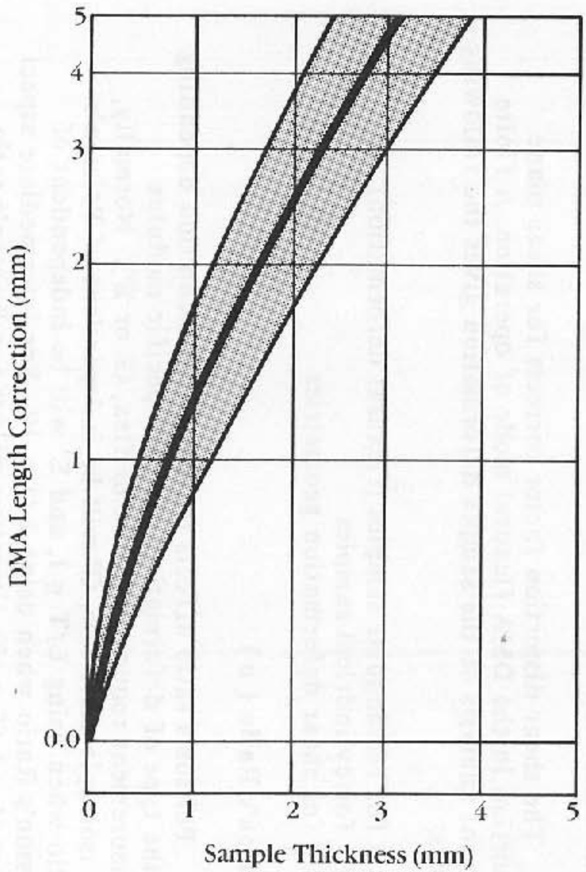
Estimated Correction: Graph I



Estimated Correction: Graph II



Estimated Correction: Graph III



Estimated Correction: Graph IV

Shear Distortion

The shear distortion factor corrects for shear plane distortion in the DMA flexural mode of operation. A finite element analysis of the sample deformation gives the following values:

- 1.5 for rectangular samples (flexural deformation)
- 1.3 for cylindrical samples
- 1.0 for shear deformation geometries

Poisson's Ratio (σ)

Poisson's ratio affects the modulus calculation depending on the type of deformation and the specific modulus measurement requested (shear or flex, G' or E'). Normally, for isotropic materials, G' will be independent of Poisson's Ratio when using $L/T \leq 1$, and E' will be independent of Poisson's Ratio when using $L/T \geq 10$. For intermediate aspect ratios ($1 \leq L/T \leq 10$), literature values for Poisson's ratio should be used if accurate modulus data is required. Similarly; in calculating G' from flexural deformation geometries or E' from shear deformation geometries, accurate Poisson's ratio data is required.

For isotropic materials, Poisson's ratio is the ratio of lateral strain to longitudinal strain for a given material subjected to uniform longitudinal stresses. In simpler terms, it is a measure of how much the sample thins down as it is stretched. Poisson's ratio will lie between 0.3 (solids and materials below Tg) and 0.5 (liquids and materials above Tg).

Some typical values for Poisson's ratio are given in Table 4.12.

Table 4.12
Typical Poisson's Ratio Values

Sample	Poisson's Ratio
Aluminum	0.334
Lead	0.431
Rubber	0.500
Carbon Steel	0.295

For anisotropic materials, for which the strict definition of Poisson's Ratio is not applicable, Poisson's ratio should be replaced by the following value:

$$\sigma = \frac{E'}{2G'} - 1$$

where E' and G' are tensile and shear moduli in the orientation axis are being investigated. For example, a unidirectional graphite fiber reinforced epoxy resin composite has a tensile modulus in the fiber axis of approximately 100 GPa due to the graphite fibers. The shear modulus in the transverse axis of the composite is the shear modulus of the epoxy resin, which can be approximated as 1/3 the tensile modulus, ca. 4/3 GPa. This gives us

$$\sigma = \frac{100}{2 \times 4/3} - 1 = 36.5$$

This high value will now influence calculated moduli at length to thickness ratios below approximately 20 for measurements in a flexural mode. Fortunately, in most practical work, aspect ratios well above 20 will be used to keep resonance frequencies below 30 Hz, so the influence of σ on calculated E' will be very minor. When working with soft anisotropic materials (e.g., fiber-reinforced rubber), which require low aspect ratios, an independently determined Poisson's ratio is necessary for accurate calculations.

Temperature Calibration

The two-point temperature calibration procedure corrects the 983's temperature, when necessary, prior to an experiment where precise transition temperatures are essential. The two-point temperature calibration should be performed after the module has been calibrated.

> **NOTE**
> **The oven temperature controller always uses the uncorrected temperature to control the sample temperature.**

Two samples with known melting points (e.g., indium and zinc) are often used to check the instrument's calibration. If the thermal scans for the samples show onset melting temperatures different from the standard values, recalibrate your instrument. The procedure for temperature calibration on the 9900 is explained in Chapter 4 of the *9900 Operator's Manual.* To perform temperature calibration on the 983 keyboard:

1. Press **TEMP CAL.**

2. Enter the observed onset melting temperature from the thermal scan for the lower temperature sample in the TEMP-1 field. Press **SHIFT** plus **RIGHT ARROW** or **ENTER** to move to the next field.

3. Enter the standard reference temperature as the CORR: (correct) temperature. For example, if you are using indium as a standard and the curve shows a melting temperature of 157.6°C, enter the information as follows:

TEMP-1:	157.60°C	CORR:	156.60°C
TEMP-2:	0.00°C	CORR:	0.00°C

4. Repeat steps 2 and 3 for the other temperature sample, TEMP-2.

Temperature calibration can be cancelled by setting observed temperature 1 and observed temperature 2 equal to zero or each other.

Modulus and Damping Equations

The equations used by the 983 DMA to calculate the complex modulus are based on the fundamental theoretical relationships in the DMA module. The derivation of these equations is explained in the paper "Basic Theoretical Relationships in DMA," presented by J.D. Lear and P.S. Gill at the 1981 NATAS Conference (see page 4-62).

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.

(1) Shear storage modulus:

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

(2) Shear loss modulus:

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

(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:

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

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

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

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

ω = $2\pi f$
M_0 = $(2J/R)\, 4\pi^2 C'V$
θ_0 = a/R
L' = effective sample length (mm),
= measured $L + \Delta L$
B = distance between pivot centers (mm)
= sample length + 2(D)
A = cross-sectional area of sample (mm^2):
$T \times W$ for rectangular samples
πr^2 for cylindrical samples
I = geometric inertial moment of sample cross section (mm^4):
$T^3 W/12$ for rectangular samples
$\pi r^4/4$ for cylindrical samples

Measured Signals

V = drive signal (mV)
f = frequency (Hz)
δ = phase angle (radians)
measured signal for fixed frequency mode;
$\pi/2$ for resonance mode

Experimental Parameters

T = thickness of rectangular sample (mm)
W = width of rectangular sample (mm)
L = length of sample (mm)
r = radius of cylindrical sample (mm)
a = 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 (mm/(mV sec^2))
K' = instrument parallel storage stiffness (N m)
K" = instrument parallel loss stiffness (N m)
= K_n" (f/fn)
f_n = frequency at which K_n" was measured (Hz)
J_C' = instrument series storage compliance (μm/N)

J_C'' = instrument series loss compliance (μm/N)

$$= \frac{J_{Cm}''\sqrt{k^2/4\pi^2}}{f_m}$$

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

These 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 after an iteration is less than one percent.

Theory of Operation

The Du Pont 983 DMA has the capability to detect mechanical relaxation processes originally studied using torsion pendulum and Rheovibron instruments (1). This is achieved by the 983's unique mechanical and electronic design which applies significantly different theoretical relationships to relate material viscoelastic constants to instrument signals and correction factors.

Basic Theoretical Relationships in DMA

All dynamic mechanical test instruments operate on specific variants of one very basic differential equation:

$$2J\left[\frac{d^2\phi}{dt^2} + D\frac{d\phi}{dt} + k^2\phi\right] = M(t)$$

Where:
- J is moment of inertia of one arm
- ϕ is angle of deformation
- D is damping coefficient
- k^2 is spring constant
- $M(t)$ is opposing moment

Each term represents a moment of force needed to overcome, respectively, the resistance to acceleration offered by the system moment of inertia (2J), energy dissipating drag forces which depend on the rate of change of the angular position variable, and elastic forces which depend linearly on the position variable itself. In a mechanical system using a static reference frame, all of these must be balanced by an opposing moment M(t) which, in the Du Pont DMA, is supplied by the motor on the driver arm.

The mechanical system coefficients D and k^2 are related to the desired viscoelastic material constants by solving the differential equation for the particular conditions imposed by the 983. These are that the motion is steady-state, the driver moment is a sine wave of amplitude M_o and angular frequency ω, and maximum displacement is fixed to $\pm\ \phi_o$ by servo-control of the driver moment.

Solution of this equation gives the following expressions for the driver moment amplitude (M_o) and phase lag (δ) of the predicted sinusoidal position wave as a function of imposed frequency and system constants:

$$M_o = 2J\phi_o \left[(k^2-\omega^2)^2 + (\omega D)^2\right]^{1/2}$$

$$\delta = \text{Arcsin} \frac{\omega D}{\left[(k^2-\omega^2)^2 + (\omega D)^2\right]^{1/2}}$$

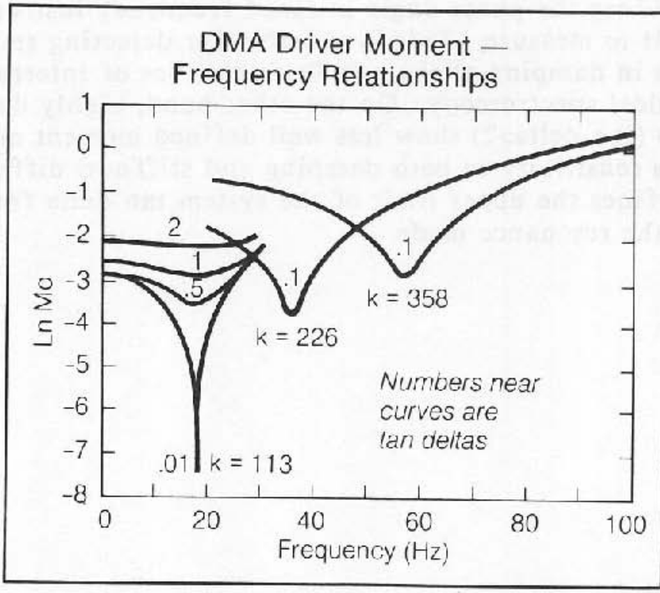


Figure 4.5
DMA Drive Moment Frequency Relationships

These equations provide a basic framework for understanding the instrument operation in that the fundamental instrument signals (M_o, ϕ, and ω) are still "uncluttered" with the sample geometrical and instrumental correction factors needed to relate the signals to sample viscoelastic constants. For example, Figure 4.5 shows the peak driver moment amplitude (in Newton-meters) required to drive systems with different "tan deltas" (system tan delta = $\omega D/k^2$) and spring constants ($2Jk^2$ in Newton-meters/radian). The driver moment exhibits a stable minimum exactly at $\omega = k$ and this minimum does not shift with tan delta. Similar calculations show the phase angle is exactly $\pi/2$ radians at the minimum. The Du Pont 983 DMA electronically "locks" this phase angle; hence the instrument running frequency is exactly equal to k (rad/sec) and can be used to calculate the elastic (storage) modulus of the sample once the deformation angle is related to sample strain through geometrical factors considered below.

The driver moment amplitude (M_o) at resonance is a very sensitive measure of system damping, particularly at small tan deltas where the phase angle in fixed frequency instruments is difficult to measure. This is valuable for detecting small changes in damping at the sub-Tg transitions of interest in mechanical spectroscopy. On the other hand, highly damped samples (tan delta>2) show less well-defined moment minima and less sensitivity to both damping and stiffness differences. This defines the upper limit of the system tan delta for the 983 in the resonance mode.

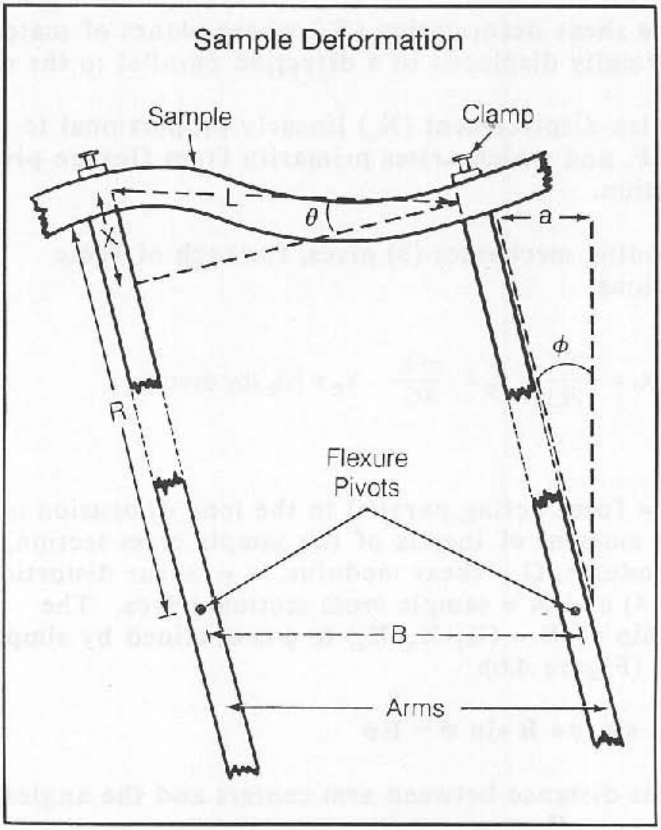


Figure 4.6
Sample Deformation

Material viscoelastic constants are related to the system parameters by expressing the sample *strain* in terms of ϕ and the sample *stress* in terms of M. The DMA arm displacement (illustrated in Figure 4.6) contains three distinct contributions:

1. Sample flexural deformation (X_f) where the arm displacement is due to a pure bending beginning at the clamp-sample junction.

2. Sample shear deformation (X_s) where planes of material are uniformally displaced in a direction parallel to the arms.

3. An extra displacement (X_c) linearly proportional to the force F, and which arises primarily from flexure pivot distortion.

Beam bending mechanics (3) gives, for each of these deformations:

$$X_f = \frac{FL^3}{12EI}, \quad X_s = \frac{\alpha FL}{AG}, \quad X_c = FJ_c \text{ (by definition)}$$

where F = force acting parallel to the long dimension of the arms, I = moment of inertia of the sample cross section, E = tensile modulus, G = shear modulus, α = shear distortion factor (3,4) and A = sample cross sectional area. The relationship of $X = (X_f+X_s+X_c)$ to ϕ is obtained by simple geometry (Figure 4.6):

$$X = L \sin \theta = B \sin \phi \sim B\phi$$

Where B is distance between arm centers and the angles ϕ and θ are small.

Sample and instrument viscoelastic quantities are related by using the *correspondence principle* (5) which allows one to formulate a viscoelastic stress response in terms of simple linear elastic moduli, to do whatever mathematical manipulations one desires, and then to substitute *complex* moduli at the end to deduce the viscoelastic response. In this case, the linear elastic equation of motion is:

$$2J \frac{d^2\phi}{dt^2} + f(\phi) = M(t)$$

where $f(\phi)$ is the elastic moment which results from the displacement. This, using the relationship of x to ϕ and including the flexure pivot spring constant is:

$$f(\phi) = \left[\frac{L^3}{12EI} + \frac{\alpha L}{AG} + J_c\right]^{-1} B^2 \phi + 2K\phi$$

This expression contains four elastic constants which are redefined as complex quantities:

E	= flexural modulus	$E^* = E' + iE''$
G	= shear modulus	$G^* = G' + iG''$
J_C	= instrument compliance	$J_C^* = J_C' - iJ_C''$
K	= flexure pivot spring constant	$K^* = K' + iK''$

where the primes define storage (') and loss (") components. Figure 4.7 depicts the series and parallel elements of this model.

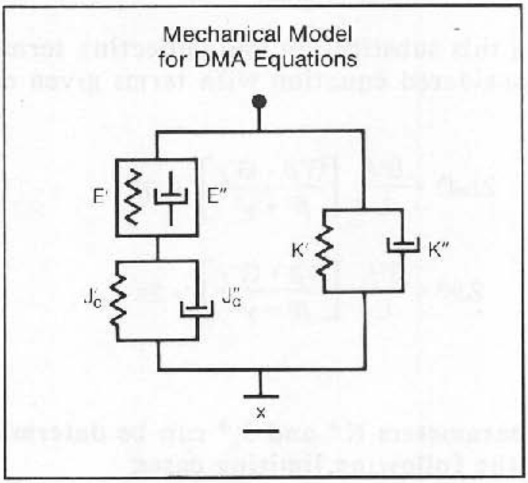


Figure 4.7
Mechanical Model

To simplify, the Poisson's ratio σ is used to relate E^* to G^*:

$$E^* = 2(1 + \sigma)G^*$$

σ is not considered a complex quantity here. To do so would complicate the analysis unnecessarily.

Substitution of the complex f (ϕ) into the differential equation and term collection gives:

$$2J\frac{d^2\phi}{dt^2} + \frac{B^2A}{L}\left[\frac{(G'\beta + G''\gamma) + i(G''\beta - G'\gamma)}{\beta^2 + \gamma^2}\right]\phi + 2(K' + iK'')\phi = M(t)$$

Where

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

$$\gamma = \frac{A}{L}\,(J_c'G'' - J_c''G')$$

For harmonic motion,

$$\phi = \phi_0 e^{i\omega t}, \quad d\phi/dt = i\omega\phi_0 e^{i\omega t} = i\omega\phi$$

Thus, the equation's imaginary part is the coefficient of

$$\omega^{-1}\, d\phi/dt.$$

Making this substitution and collecting terms gives the originally considered equation with terms given explicitly by:

$$2J\omega D = \frac{B^2A}{L}\left[\frac{G''\beta - G'\gamma}{\beta^2 + \gamma^2}\right] + 2K''$$

$$2Jk^2 = \frac{B^2A}{L}\left[\frac{G'\beta + G''\gamma}{\beta^2 + \gamma^2}\right] + 2K'$$

Instrument parameters K^* and J_c^* can be determined by considering the following limiting cases:

1. G', G" = 0 (no sample)

Then

$J(\omega D)_o = K''$ (Parallel loss stiffness)
$Jk^2_o = K'$ (Parallel storage stiffness)

2. $G' = \infty$, $G'' = 0$ (very stiff, loss-free sample, e.g., steel)

$$2J(\omega D)_{\infty} = \frac{J_c'' B^2}{|J_c^*|^2} + 2K'' \text{ (series loss compliance)}$$

$$2Jk^2_{\infty} = \frac{J_c' B^2}{|J_c^*|^2} + 2K' \text{ (series storage compliance)}$$

For the resonance mode of operation, ϕ is fixed at a/R and δ is fixed at $\pi/2$ radians. The equations of motion then require that $k = \omega = 2\pi f$ where f equals the measured frequency in Hz. When this is the case, $\omega D = M_0/2J\ \phi_0 = 4\pi^2 C'V/a$, where V is the millivolt damping signal from the instrument and C'/a is the drive signal conversion factor determined by the instrument calibration procedure. The equations for storage and loss moduli are:

$$G' = (2Jk^2 - 2K')\frac{\beta^2 + \gamma^2}{\beta}\ \frac{L}{B^2 A} - \frac{\gamma G''}{\beta}\ ,\ E' = 2(1+\sigma)\,G'$$

$$G'' = (2J\omega D - 2K'')\frac{\beta^2 + \gamma^2}{\beta}\ \frac{L}{B^2 A} + \frac{\gamma G'}{\beta}\ ,\ E'' = 2(1+\sigma)\,G''$$

Since these are quadratic in the moduli (β and γ are linear in G' and G"), it is best to iterate a solution for G' and G" using $\gamma = 0$ as a first approximation.

Table 4.13 illustrates this for hypothetical samples stiff enough to give a significant instrument compliance correction with different assumed tan deltas.

Table 4.13
Example Calculations

Example Calculations

Parameters Used

	J	L	T	W	B	σ	K'	K"	J'_c	J''_c	a	C'
Value	1.5	20	1	10	32	.5	.33	0	1.4E-6	6.7E-8	0.2	.052
Units	g-m²	← mm →					Nm		mN⁻¹ →		mm	sec²/mV-mm

Assumed		Computer-Calculated					Equations-Calculated					
G'	Tan δ	β	γ	f (π/2)	M_θ (π/2)	mv*	G' (1st order)	Tan δ (1st order)	G' (2nd order)	Tan δ (2nd order)	G' (3rd order)	Tan δ (3rd order)
1.E11	.01	204.4	-2.65	46.0	.009	224	.66E11	.023	1.E11	.01	1.E11	.01
1.E11	.05	204.5	0.15	46.0	.015	365	.66E11	.045	1.E11	.05	1.E11	.05
1.E11	.10	204.7	3.65	46.0	.025	584	.66E11	.072	1.E11	.1	1.E11	.1
1.E11	.50	206.0	31.65	47.1	.111	2703	.69E11	.316	1.08E11	.47	1.E11	.5
1.E11	.75	206.8	49.1	48.2	.16	3897	.72E11	.436	1.16E11	.67	1.E11	.75
1.E11	1.0	207.7	66.6	50.1	.20	4895	.78E11	.507	1.30E11	.83	1.E11	.99

1st order: $J'_c, J''_c = 0$

Coupled Equations: First Iteration (2nd order); Second Iteration (3rd order)

$^*mv = \dfrac{M_0 a}{2J\theta_0 4\pi^2 C'} = 2.44 \times 10^4$ M_0 for J = 1.5 g m², $\theta_0 = a/150$

Clamping Corrections

All mechanical methods for measuring material viscoelastic constants require careful consideration of clamping corrections. In tension measurements on stiff samples, large clamping errors are common because the clamp forces are perpendicular to the deformation driving force. Because of this, torsion or flexure is often preferred where the driving force is directed primarily toward the clamping support surfaces. Flexure in particular simulates many end use conditions, but calculated moduli are sensitive to length corrections. Since these are difficult to determine in any given situation, experimental methods are needed. Massa (6) has defined these for the Rheovibron, and Read and Dean (7) for torsional and some flexural instruments. For the specific case of the Du Pont instrument, the basic procedure of Read and Dean is followed in making the substitution $L + \Delta L$ everywhere for L in the mechanical equations.

For rectangular samples, the equation which results is:

$$G\,(\Delta L = 0) = G\left\{1 - \frac{\Delta L}{L}\left[\frac{2(1+\sigma)T^2\alpha + 3L^2}{2(1+\sigma)T^2\alpha + L^2}\right]\right\}$$

$$\text{where } \frac{1}{L}\left[\frac{2(1+\sigma)T^2\alpha + 3L^2}{2(1+\sigma)T^2\alpha + L^2}\right] = \xi$$

(Valid for $\Delta L/L \ll 1$)

and where $G(\Delta L = 0)$ is the value calculated using $\Delta L = 0$ and G is the "true" value. Plotting $G(\Delta L = 0)$ versus ξ affords G as the $L^{-1} = 0$ (infinite length) intercept and allows ΔL to be calculated from the slope. This is illustrated in Figure 4.8 for a polycarbonate sample, showing the linear correlation between shear modulus and ξ in the equation above. Two-dimensional finite element analysis indicates that the length correction should depend on the extent of sample compression within the clamping region. About 1.5% compression compensates for slippage-inducing tensile stress at the sample-clamp boundary surface. For accurate work, however, experimental determination of clamp corrections is advisable since clamp geometry is seldom perfect and, especially for smooth surface, high modulus samples, adequate compression within the clamps may not be possible.

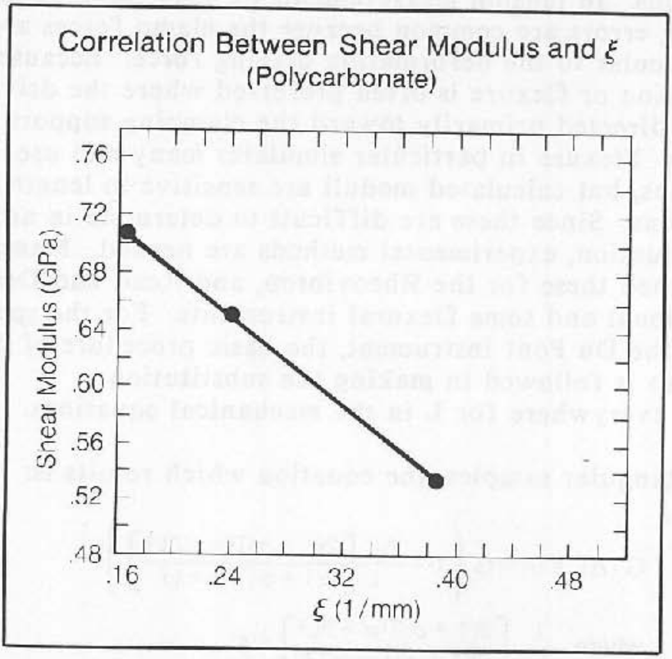


Figure 4.8
Correlation between Shear Modulus and ξ

The correction factors discussed in this paper are summarized in Table 4.14.

Table 4.14
Correction Factors

Item	Benefit	Where Significant	Typical Values
Length Correction -End effects for jaw clamped samples	Reproducible and quantitative modulus	Short, hard samples	-0.5 to +2 mm
Instrument Compliance -Non-rigid system	Quantitative modulus values	Stiff samples frequencies>30Hz	1 μm/N
Instrument Damping -Mechanical friction	Improved damping measurements	Low damping, high frequency	0.1 N m; 0.05 μm/N
Instrument Stiffness -Spring constant	Ability to run supported liquids	Low frequency soft samples	0.36 N m
Poisson's Ratio	Mixed deformation (flexure/shear analysis)	Aspect ratio (L/T) between 2 and 8	0.5 (Rubbery) 0.33 (Glassy)
Shear Distortion	Quantitative accuracy for modulus	Short samples L/T<10	1.5 Rectangular samples 1.3|Cylindrical samples

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5. D.R. Bland, The Theory of Linear Viscoelasticity, Oxford: Pergamon (1960).

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Troubleshooting Guide

SYMPTOM		PROBABLY CAUSED BY		CORRECTION
Frequency does not stabilize (resonance mode).	1.	Sample is too soft for clamping arrangement.	1.	Decrease gap distance (< 10 mm).
	2.	Sample has high modulus and gap distance is too small.	2.	Increase gap distance (> 40 mm).
	3.	DMA is not on a stable surface.	3.	Move DMA to vibration-free bench.
Driver motor does not start (Error 83 or 84).	1.	Locking pins have not been removed.	1.	Remove locking pins prior to starting run.
	2.	Sample (especially soft material) is affected by stray air movement.	2.	Place heater assembly on the DMA.
	3.	Sample is very soft.	3.	Increase oscillation amplitude to at least 0.40 mm.
Gain does not stabilize (fixed frequency mode).	1.	Low loss sample is being run above its resonant frequency.	1.	Choose a frequency below resonance.
			2.	Run at a higher oscillation amplitude.

Frequency, drive signals and phase angle (fixed frequency mode) show excessive noise or spikes.	1. Slide lock on DMA front panel is not fully tightened.	1. Tighten slide lock prior to run.
	2. DMA is not located on a stable surface. surface.	2. Located DMA on a vibration free
	3. Sample is slipping.	3. Clamp sample (below its glass transition temperature) to a torque of 8 in lbs or greater.
	4. Thermocouple is touching sample.	4. Move thermocouple back such that there is 1 mm between Tc tip and sample.
Sample exhibits excessively high loss modulus, E" or tan delta.	1. DMA has not been calibrated for phase angle.	1. Ensure that phase angle calibration has been performed, especially when operating at fixed frequencies greater than 0.5 Hz.
	2. Sample is oscillated at a fixed frequency which is greater than sample resonance frequency.	2. Decrease fixed frequency to 2.0 Hz or less.

Sample loses oscillation during run.	1. DMA is located on a non-stable surface.	1. Place DMA on vibration-free bench.
	2. Oscillation amplitude is not sufficiently large enough to properly drive the sample.	2. Increase oscillation amplitude.
Large damping signal is obtained when calibrating for compliance factor (Jc') using thick steel bar (damping signal > 1400 mv).	1. DMA is located on non-stable surface.	1. Move DMA to vibration-free bench.
	2. Felt pads have been removed from DMA feet.	2. Replace felt pads on all 3 DMA feet.
	3. Bolts holding down upper plate on electronics housing have come loose.	3. Tighten all four bolts (Note: Two bolts are located under driver housing).
	4. Build-up of residue clamp faces.	4. Remove residue using appropriate solvent.
	5. Oscillation amplitude is set too high.	5. Decrease the oscillation amplitude to 0.10 mm.
	6. Heater assembly is not in place.	6. Place the heater assembly on DMA and ensure that both bolts have been tightened.

Negative length corrections are obtained on stiff samples.	1. Series compliance factor Jc' is too large (Jc' should be less than 1.5 μm/N).	1. Redo calibration for Jc' and ensure that a frequency of 85 to 90 Hz is obtained.
	2. Sample is slipping in the clamps.	2. Wrap the ends of sample with either a single layer of thin masking tape or aluminum foil.
Modulus values are not correct.	1. Length correction has not been performed.	1. Do the length correction measurement and enter it into the 983.
	2. Sample dimensions have not been properly measured.	2. Measure the width and thickness prior to mounting the DMA. Measure the sample length after the sample is clamped in the DMA.
	3. Correct value for "clamping distance" has not been entered.	3. Ensure that the clamping distance is 8.0 ± 0.2 mm.
	4. Calibration factors are not correct.	4. Recalibrate the 983 DMA.

Modulus values are not correct. (cont'd)	5. The sample being analyzed is an isotropic (i.e., uniaxially or biaxially oriented). For these materials (such as composites), the DMA flexural modulus cannot be compared to a tensile modulus.	5. Run the sample on an Instron in a bending mode of deformation for a more reliable comparison.
	6. Sample has been distorted during mounting in the DMA clamps.	6. Cool the sample below its Tg, and then tighten clamps locking screws.
"Gain error" occurs during a run.	1. Power required to drive sample exceeds the upper limit.	1. Decrease obtained oscillation amplitude or increase sample length.
	2. Sample expansion or contraction has caused the sum of the drive signal and offset signal to exceed the motor power limit.	2. Decrease oscillation amplitude, increase sample length, or change the adjustment of the slide lock to decrease the offset signal.
		3. Stop the motor. Use the length adjustment to zero the sample position and restart the motor.

CHAPTER 5

Maintenance and Diagnostics

Maintenance and Diagnostics

Maintenance

You should perform routine maintenance to keep the 983 in good working condition. The procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of Du Pont Instruments or other qualified service personnel.

> **WARNING**
>
> **Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.**

The mechanical parts of the 983 should be inspected and cleaned periodically.

Inspection

Examine the pivots, thermocouple, and oven for good condition as follows:

Pivots - The oscillating arms must have free lateral movement. Even the slightest restriction in movement will reduce test accuracy.

Thermocouple - The thermocouple must be mounted firmly in the mounting block, and the tubes must not be dented or cracked. The electrical connections for the thermocouple must be complete.

Oven - The rim seal of the oven must be smooth to make a tight seal when the oven is installed.

Cleaning

Test result accuracy depends on how well the pivots function. Clean the pivots periodically as follows:

Using low air pressure, 175 kPa (25 psig) maximum, blow accumulated materials away from the pivot area. Steady both arms while applying air pressure.

Spray a cleaning solvent, such as Freon(R), in and around the pivots. Use a generous amount of cleaning solvent, but do not flood the area.

Keyboard

You can clean the local keyboard as often as you like. The keyboard is covered with a silk-screened Mylar(R) overlay that is reasonably water resistant but not waterproof or resistant to strong solvents or abrasives.

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

Lubrication

Spray a lightweight lubricant on the slide surface of the mechanical slide to protect the steel parts when running below ambient and opening while cold. Wipe off excess lubricant.

Locking Pin Adjustment

The locking pins should be checked and adjusted periodically as follows:

1. Check to see that the locking pins are in the arms securely so that a little force is needed to pull them out. The locking pins should not move when in the locked position. If the locking pins are too tight, you will not be able to remove them from the arms.

2. Turn the adjustment screw as shown in the figure below to tighten or loosen the locking pins.

NOTE

If the locking pins are installed so tightly that they cannot be removed, loosen the setscrew and then remove the pins.

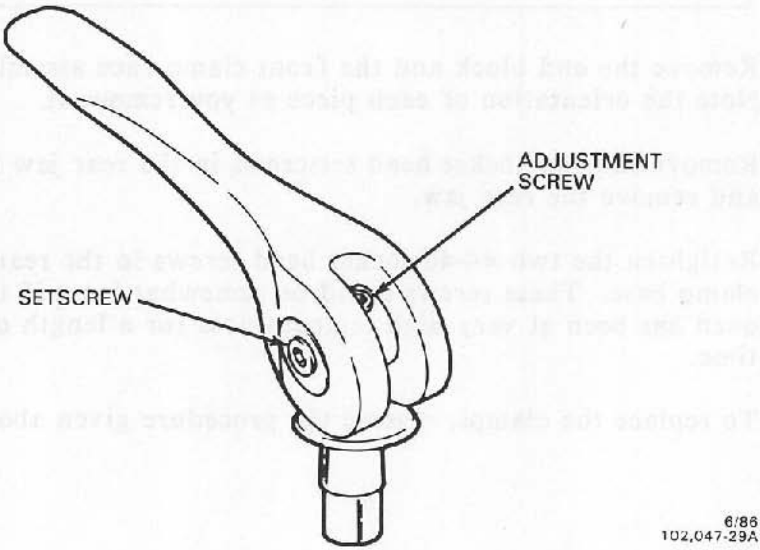

Figure 5.1
Locking Pin

Clamp Mounting

Vertical Clamps

The following procedure applies to smooth, serrated, and notched clamps. To change clamps:

1. Install the locking pins in both arms.

CAUTION

The thermocouple ceramic is fragile and can easily be damaged. It is recommended that the thermocouple be moved out of the way before changing clamps.

2. Remove the two rear nuts on the end of each clamp. There is a small spring washer behind each nut.

NOTE

The wrenches needed for clamp removal are included in the 983 accessory kit.

3. Remove the end block and the front clamp face assembly. Note the orientation of each piece as you remove it.

4. Remove the two socket head setscrews in the rear jaw face and remove the rear jaw.

5. Retighten the two #4-40 socket head screws in the rear clamp base. These screws could be somewhat loose if the oven has been at very high temperatures for a length of time.

6. To replace the clamps, reverse the procedure given above.

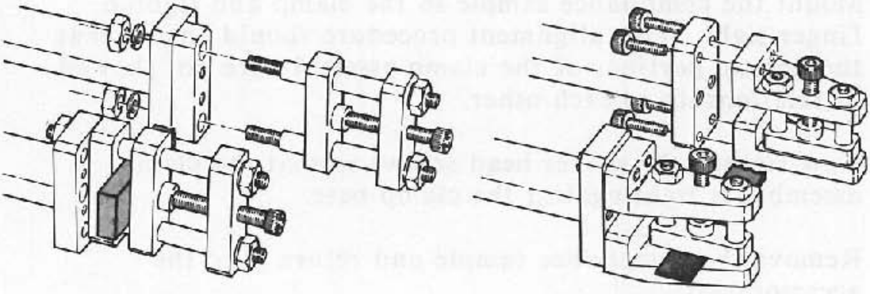

Figure 5.2
Exploded View of Clamps

> **NOTE**
>
> **When installing notched clamps, make sure that notches of the same size meet (front and back), and that the orientation of each set of clamps is the same.**

Horizontal Clamps

1. To remove the vertical clamp assembly, follow steps 1 through 5.

> **NOTE**
>
> **The compliance sample and the tools needed for removal and installation of the clamps are located in the 983 accessory kit.**

2. Install the horizontal clamp assembly onto clamp base by using the four socket head screws supplied with each half of the clamp assembly. Leave these screws partially loose so the clamps can be aligned.

3. Mount the compliance sample in the clamp and tighten finger-tight. This alignment procedure should ensure that the bottom portions of the clamp assembly are not skewed in relationship to each other.

4. Now tighten the socket head screws so that the clamp assembly is tight against the clamp base.

5. Remove the compliance sample and return it to the accessory kit.

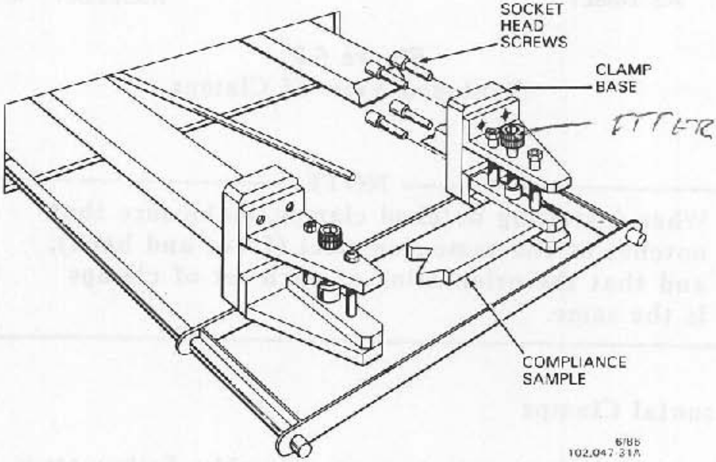

Figure 5.3
Horizontal Clamp Alignment

Constant Tension Springs

The DMA constant tension springs are designed for subambient elastomeric sample experiments. As the sample shrinks with decreasing temperature, the springs flex to compensate and the sample is retained. This eliminates the need to remove the DMA head to retighten the clamps at subambient conditions. The springs work best when used with serrated jaw clamps, although they may be used with any of the vertical clamps.

The constant tension springs are included in the accessory kit.

1. Install the vertical serrated clamps following the procedure previously described.

2. Insert one spring in each arm assembly between the outer jaw clamps and the clamp locking screw. See Figure 5.4. The notches in the spring should slide along the jaw mounting rails.

3. Mount the sample following the usual procedures.

4. Turn the clamp locking screw until it just touches the spring, then tighten the screw seven turns to obtain a clamping force of 4.2 inch-pounds on the sample. (The spring rate is equal to 0.6 inch-pounds per screw turn.)

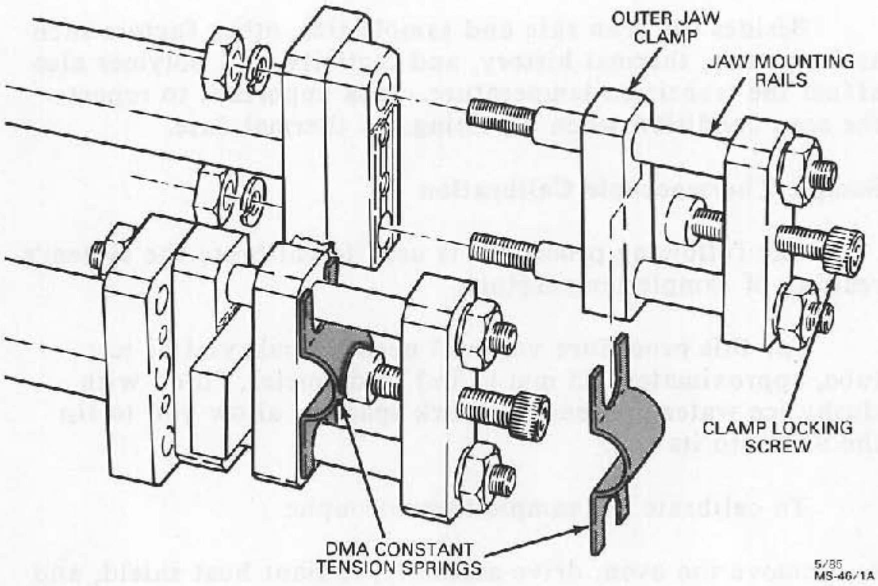

Figure 5.4
Constant Tension Springs

Temperature Calibration

The 983 DMA temperature calibration is based on an isothermal thermocouple adjustment using an ice bath. Sample temperature lags occur at high heating rates (>5°C/min) and/or with thick sample (>3 mm). This phenomenon should either be minimized (smaller sample size and/or slower heating rate), or its effect should be corrected using the Thermal Lag Device supplied in the DMA accessory kit. A temperature correction can also be applied using the temperature calibration routine to calibrate the system using a reference standard (see Chapter 4).

The polycarbonate sample supplied in the 983 accessory kit, while not a standard reference material, gives reproducible Tg at a damping peak of 153°C. This may be used to indicate the sample thermocouple versus heating effects and provide the above correction.

Besides the scan rate and sample size, other factors such as frequency, thermal history, and tacticity of a polymer also affect the transition temperature. It is important to report the scan condition when reporting the thermal date.

Sample Thermocouple Calibration

The following procedure is used to calibrate the system's reading of sample temperature.

For this procedure you will need a small vial or test tube, approximately 25 mm (1 in.) in diameter, filled with slushy ice water and enough work space to allow you to tip the 983 onto its end.

To calibrate the sample thermocouple:

1. Remove the oven, drive assembly, radiant heat shield, and sample tray.

2. Install the locking pins into the arms.

3. If there is a sample installed, remove it. Leave the locking pins in place.

4. Open arm spacing to 50 mm (2 in.).

5. Tip the 983 onto its right end so the arms face downward.

CAUTION

The thermocouple ceramic is very delicate and may be broken if handled roughly.

6. Gently slip the ice bath around the thermocouple. Be careful not to bump or bend the thermocouple.

7. Hold the ice bath on the thermocouple and record the temperature value. If the temperature error exceeds ±5°C, call Du Pont service to have the thermocouple recalibrated. Otherwise, use the two-point temperature calibration function to correct the temperature. See Chapter 4.

After the calibration is complete, readjust the position of the thermocouple as described in the sample mounting procedures on page 3-11 of this manual.

Thermal Lag Device Installation

This device helps to correct for thermal lag in samples of low thermal conductivity and thicknesses of 5 mm or more. The end of the lag device should be positioned the same as the sample thermocouple would be without the device, one-third of the sample width from the top of the sample and one-third of the sample length from the driven arm.

NOTE

If you are an owner of an upgraded 982 and choose to use the thermal lag device, you must have the new thermocouple bracket (PN 982221.901) installed on your 983. This bracket is shown in Figure 5.5. If your unit does not have the bracket shown, contact Du Pont Sales Office (302-772-5500) to request the correct bracket.

To install the thermal lag device:

1. Loosen the thermocouple hold-down screw so that the thermocouple's ceramic sleeve can move within the bracket.

2. Slide the thermal lag device over the thermocouple and the black metal extension until it meets the bracket face.

CAUTION

If the thermocouple sleeve is not loose, the thermocouple may break.

3. The thermocouple tip should now be in contact with the inside front face of the lag device. Hold the lag device in place while moving the thermocouple ceramic sleeve away from the lag face approximately 5 mm (0.18 in.).

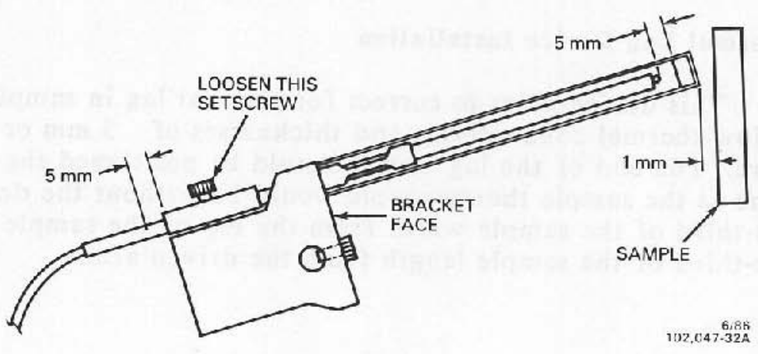

Figure 5.5
Thermocouple Bracket

Diagnosing Power Problems

Fuses

There are several internal fuses; however, NONE are considered user serviceable. If any of the internal fuses blow, a hazard may exist. Call Du Pont for service.

The only fuses that you should service yourself are located on the module control panel. Both fuses are housed in safety-approved fuse carriers labelled F1 and F2. See Figure 5.6.

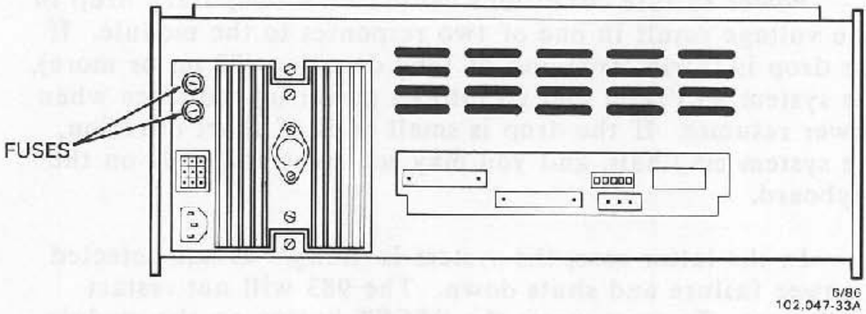

Figure 5.6
Fuse Locations

> **WARNING**
> **Always unplug the instrument before you examine or replace the fuses.**

Fuse F1 is in the circuit between the main electrical input and the rocker switch labelled "Power." All power for internal operations and module functions, except heater power, passes through this fuse. If this fuse blows, you will get no response from the system.

Fuse F2 protects the heater coils in the oven, the block heaters in the drive assembly, and supplies power to the optional LNCA.

Because F2 does not power the internal logic, you may not know that this fuse is blown until you try to heat a sample, since the 983 module will pass its confidence test with this fuse open.

The fuse is checked whenever a method is beginning. Power supplied by this circuit is switched by a computer controlled relay as well as the "HEATER" switch located on the 983 DMA front panel. When both devices are active the lamp in the "HEATER" switch will turn on.

Power Failures

Power failure conditions caused by a temporary drop in line voltage result in one of two responses to the module. If the drop is fairly large and of long duration (20 ms or more), the system will reset and go into its power-up sequence when power resumes. If the drop is small or is of short duration, the system may halt, and you may see error code F02 on the keyboard.

In the latter case, the system is "hung." It has detected a power failure and shuts down. The 983 will not restart until reset. To reset, press the **RESET** button on the module base back panel or on the keyboard.

If the "power fail" error (code F02) appears at start-up and remains even after you have tried to restart the instrument, the detection circuitry itself is probably at fault. Do not try to repair it yourself; call Du Pont for service.

The 983 module is designed for a nominal line voltage of 115 V ac, 50 or 60 Hz. The module should not be operated at less than 105 V ac or greater than 130 V ac. Low line voltage may result in poor module operation; high line voltage may damage the instrument.

983 DMA Test Functions

The 983 has two levels of test and diagnostic functions: 1) confidence tests that are run every time the instrument is started; and 2) TEST key functions that can be used to monitor various functions once the 983 is up and running.

These test functions are always present in the instrument. They are designed to aid both manufacturing and service in checking out and repairing the 983. The TEST key functions are controlled by the keys on the keyboard/display unit. All the tests display status and error information on the keyboard/display unit.

Confidence Test

The 983 Confidence Test is run each time the DMA is turned on or reset. The confidence test checks most of the computer and interface components in the system.

```
Ver 2.0   983 DMA Confidence Test   60
```

6/86
102,047-34A

Figure 5.7
During the Confidence Test

Any errors are reported on the keyboard/display unit. Non-fatal errors are displayed for three seconds, and then the confidence test continues with the next test.

The confidence test can be cycled indefinitely by selecting the confidence test option (option 8) from the **TEST** key. When run in the cycle mode, the test will halt on any error. This allows for unattended testing since the error message will be displayed on the keyboard until the system is reset.

```
Ver 2.0   983 DMA Confidence Test     60
Main DRAM error   Rd2553 WrA553 XOR8000
```

8/86
102,047-34A

Figure 5.8
Error Message

When running, the current confidence test number is on the keyboard/display. The test number appears as a two-digit hex number at the upper right on the display. See Figure 5.7. This number is changed as each new test is started. Most of the tests are very quick, so their test numbers may not be apparent. If an error is detected, an error message is posted on the bottom line of the display. See Figure 5.8. If the error is not fatal, the program will pause at the error display for 3 seconds before going on to the next test. The length of time required to run the confidence test depends on the options installed. The base level system takes about 3 seconds, while a fully configured system takes about 5 seconds. The longest tests are the RAM board tests, which take about 1.5 seconds each. After the tests are completed, a sign-on message is displayed for 3 seconds. The system then starts running, and the **READY** light on the back of the 983 is turned on. See Figure 5.9.

Fatal error means that a circuit essential to the operation of the 983 has failed the confidence test. The module cannot reliably perform any further functions. The fatal error message is posted on the keyboard display, and the system stops. The **READY** light will remain off.

```
128K        983 DMA Module        GPIB-1
Copyright 1986 E. I. DuPont Company Inc.
```

6/86
102,047-11A

Figure 5.9
Sign-on Screen

Table 5.1
Primary Confidence Test and Error Code Summary

30	CMOS ram read/write test	(fatal)
40	PROM checksum, location, and version test	(fatal)
5n	CPU board I/O function tests	(fatal)
60	Memory module (DRAM) read/write test	
61	Memory expansion module read/ write test	
70	Keyboard shorted key test	
80	Display read/write test	
90	GPIB board test	
B0	CMOS saved memory checksum test	

Confidence Test Descriptions

30 - CMOS memory read/write test.

This test checks the CMOS RAM by filling the memory with a pseudo-random bit pattern, reading it, complementing the values, and reading them back again. Any mismatch between the written and read data is reported on the display.

Wr = Value written into memory
Rd = Value read back from memory
XOR = Exclusive or of the written and read values

The "XOR" value identifies which bits were in error. Bits in the two low digits are located in the low CMOS chip (MEM-4, U48); bits in the upper two digits are located in the high CMOS chip (MEM-5, U52). A second part of the RAM test checks the even/odd byte addressing hardware. This test is like the first test except it reads and writes bytes instead of words. Since the RAM chips have already been tested, any errors at this stage are due to problems in the even/odd byte addressing hardware. This error is reported as a 'Byte Addressing error.' All CMOS RAM errors are FATAL; the 983 cannot run without the CMOS memory.

40-47 - Program PROM checksum, location, and version test

These tests check the checksum, software version level, and location code for each of the program PROMS (MEM-0, MEM-1, MEM-2, MEM-3, MEM-6, and MEM-7). Errors are reported on the display with the PROM location (MEM-n) and the type of error. All program PROM errors are fatal.

48 - Option PROM checksum

This test checks the option PROM (U65). Any error is reported as a 'bad software option PROM' and the option level is set to the lowest option (resonance only). The system will continue to run with a bad option PROM.

50 - Waveform counter test (U7)

This test checks the functionality of the 9513 chip that measures the arm position, oscillation amplitude, oscillation frequency and phase angle and controls the fixed frequency waveform. This chip is essential to controlling and monitoring the sample during an experiment. Any errors are fatal.

51 - Temperature control counter test (U27)

This test checks the functionality of the 9513 chip that measures the sample and oven temperature, the heater power level, and controls the TRIAC firing angles for the oven and LNCA heaters. This chip is essential to control the temperature during an experiment. Any errors are fatal.

52 - Watchdog timer test

The watchdog timer circuit is used to prevent the CPU from getting hung up in a state where it will not respond to I/O interrupts. The timer also runs the "ready" light on the back of the 983. The ready light will glow briefly when the timer is being checked. All watchdog timer errors are fatal.

6n - Data storage memory (DRAM) read/write test

These tests check the memory module and RAM expansion (DRAM) boards. These boards contain the local data (run) storage memory. This test is the same as the CMOS memory test; it fills the memory with a pseudo-random bit pattern, reads it, complements the values, and reads them back again. Any mismatch between the written and read data is reported on the display.

Main DRAM	= Memory module board
Upper DRAM	= RAM expansion board
Wr	= Value written into memory
Rd	= Value read back from memory
XOR	= Exclusive or of the written and read values

The "XOR" value identifies which bit(s) was in error. Each bit is in a separate 1x64K RAM chip, so the faulty chip(s) can be located. The memory module (if present) is checked first, followed by the expansion board (if present). A second part of the RAM test checks the even/odd byte addressing hardware. This test is like the first test except it reads and writes bytes instead of words. Since the RAM chips have already been tested, any errors at this stage are due to problems in the even/odd byte addressing hardware. This error is reported as a 'Byte addressing Error.' Any errors will cause that board to be declared "offline" and unavailable for use. The system will run without the DRAM boards.

70 - Keyboard shorted key test

This test scans the keyboard and looks for key closures. If any of the keys are shorted (or were being pressed during the test), a 'shorted key' error message is displayed. The system will continue to run.

80 - Display read/write test

This test checks the I/O lines between the 983 and the keyboard/display unit by writing to the display and then reading the data back. Any errors are reported with the data.

Wr = Byte written to the display
Rd = Byte read from the display

Most display errors will be obvious from the garbled messages that appear on the display. The system will continue to run with a bad display.

90 - GPIB board test

This test checks the functionality of the GPIB talker/listener chip on the GPIB board. Any errors cause the GPIB to be declared unavailable. The system will continue to run, but will not communicate with the 9900.

B0 - CMOS saved memory checksum test

This test checks the checksum for the saved memory region of the CMOS memory. An error normally indicates a problem either in the power fail circuit or the battery power circuit for the CMOS chips. The checksum is recalculated and an 'Err 15' is reported when the system starts running.

Test Functions

Ten test functions are available to test the 983 Dynamic Mechanical Analysis System. The test options are:

1. Signals - displays a variety of signals pertinent to the current motor mode. The top line displays the measured frequency or phase; absolute arm position or center of oscillation; and the measured oscillation amplitude or relative arm displacement. The second line contains the oscillation symmetry; the offset DAC output and attenuation; and the gain DAC output and attenuation. Most of these signals are available in a more readable form from the **STATUS** key on the keyboard or the **Signal Control** page on the 9900.

2. Display - fills the entire display screen with a single character to test the function of each cell.

3. Display - tests the character set.

4. Not used.

5. MRD - reads from specified memory locations. For Field Service use.

6. MWR - writes into specified memory locations. For Field Service use.

> **CAUTION**
>
> **This test may cause system failures if used improperly.**

7. Confidence Test Error Readout. For Field Service use.

8. Confidence Test Cycle - initiates a continuous confidence test cycle. To escape from the test, press RESET.

9. Motor Test - manually controls the motor zeroing relay. For Field Service use.

10. Heater Power V/F Frequency - displays the frequency from the heater power measurement circuitry. For Field Service use.

Refer to the Service Manual or contact Field Service for further information.

Appendix

Nonfatal Error Codes

The nonfatal error codes, the error message, and interpretations are summarized below. Contact Du Pont service for further assistance.

Err 10 **Confidence test error during start up. Contact Field Service.**

Problem: This is a general message, which indicates that some nonfatal error has occurred and a portion of the system is faulty.

Solution: If you find that your work is not affected by the malfunctioning part of the system, continue your experiment and call Du Pont for service.

Err 12 **GPIB bus error. Bus controller not listening.**

Problem: The GPIB control line is faulty. The CPU was able to communicate with the controller, and it now has a message to send; however, it has not received an acknowledgement. This is a nonfatal error.

Solution: If the problem does not affect your work, continue with your operation and then call Du Pont for service.

Err 15 **CMOS RAM check sum error. Stored parameters lost.**

Problem: This problem indicates that the values in the save memory region were corrupted when the power was last turned off.

Solution: Turn the power off and then on again. If the error is recreated, call Du Pont for service.

NOTE

This error will occur the first time the unit is turned on after the 983 module software is revised.

Err 21 **Run Number already exists. Delete previous run or change number.**

Problem: The given run number already exists. This usually occurs in the SAMPLE ID section.

Solution: Delete the stored run in memory or enter a different run number.

Err 25 **Input value less than lower limit.**

Problem: The value entered is less than the system requires for that particular parameter.

Solution: Use the **DEC** key to find the limit and enter a number within that range.

Err 26 **Input value greater than upper limit.**

Problem: The value entered is greater than the system requires for that particular parameter.

Solution: Use the **INC** key to find the limit and enter a number within the range.

Err 27 **Invalid number or illegal input character for this parameter type.**

Problem: Text has been entered into a numeric field, or an invalid Yes/No response has been input.

Solution: Type valid characters into the field and press **ENTER**.

Err 39 **(String from external computer)**

Problem: This message was sent by the 9900 computer.

Solution: Refer to the 9900 manual for assistance.

Err 40 **GPIB Invalid command.**

Problem: A problem exists with the communication hardware or software.

Solution: Call Du Pont for service.

Err 41 **GPIB Input value too large.**

Problem: A problem exists, with the communication hardware or software.

Solution: Call Du Pont for service.

Err 42 **GPIB Input value too small.**

Problem: A problem exists with the communication hardware or software.

Solution: Call Du Pont for service.

Err 43 **GPIB Bad numeric input string.**

Problem: A problem exists with the communication hardware or software.

Solution: Call Du Pont for service.

Err 44 **GPIB String too long or no ETX.**

Problem: A problem exists with the communication hardware or software.

Solution: Call Du Pont for service.

Err 45 **GPIB Real-time or playback data buffer overflow. Too many points.**

Problem: This problem exists in the communication software. It is not user caused.

Solution: The system will automatically correct the problem.

Err 46 **GPIB Cannot modify or delete the running method.**

Problem: The system is set up so that you cannot change a method while it is in progress.

Solution: Wait until the run is completed, and then change or delete the method.

Err 47 **GPIB Insufficient room in memory for method. Delete an existing method.**

Problem: There is not enough memory left in the module for the method you are programming.

Solution: Use Method Editor to delete an existing method.

Err 48 **GPIB Method segment out of order.**

Problem: A problem exists with the communication hardware or software.

Solution: Call Du Pont for service.

Err 49 **GPIB cannot delete an active run.**

Problem: The system is set up so that you cannot delete a run's data while the data is still being acquired.

Solution: Use the **REJECT** key to delete an active run or wait until the run is completed.

Err 50 **Previous segment not defined.**

Problem: This error occurs during Method editing, when trying to edit a segment beyond the end of the method.

Solution: Go back to the first segment. Use the **DOWN ARROW** or **INC** key to find the desired segment, type in the parameters, and press **ENTER**.

Err 51 **Cannot change segment type while method is running.**

Problem: The system is set up so that you cannot change a method segment type while that particular method is running.

Solution: Wait until the run is completed; then change the segments.

Err 52 **Cannot insert new segment while method is running.**

Problem: The system is set up so that you cannot insert or add a new segment to a running method.

Solution: Wait until the run is completed; then make your changes.

Err 53 **Cannot delete segment while method is running.**

Problem: The system is set up so that you cannot delete segments in the running method.

Solution: Wait until the method is completed; then make your changes.

Err 54 **Cannot delete running method.**

Problem: The system is set up so that you cannot change a method while it is in progress.

Solution: Wait until the run is completed; then delete the method.

Err 55 New method number cannot be the same as the source method.

Problem: You are trying to copy a method to itself.

Solution: Choose a unique method number for the new method created by the copy method function.

Err 56 Source method is empty.

Problem: There are no segments for the method you have designated as the source method.

Solution: Select a different source method.

Err 57 No more method segments available. Delete an existing segment.

Problem: All 60 segments were used for methods already set up in the system; no more segments are available.

Solution: Delete or edit a method to free up additional segments.

Err 60 Run method is empty. Cannot start run.

Problem: You have not defined the segments for the method number you are trying to run.

Solution: Use the **METHOD** key to define a method for the experiment or select a method number with an existing method.

Err 62 Segment Final Temp greater than upper limit. Cannot start run.

Problem: The final temperature of a segment in the method program exceeds the module's upper limit of 500°C.

Solution:	Use the Method Editor to change the segment's final temperature.
Err 63	**Segment Final Temp less than lower limit. Cannot start run.**
Problem:	The final temperature of a segment in the method program exceeds the module's lower limit of -200°C.
Solution:	Use the Method Editor to change the segment's final temperature.
Err 64	**Cannot modify segment. Method is not running.**
Problem:	The **MODIFY SEGMENT** key functions only when a method is running.
Solution:	Start the experiment; then use the **MODIFY SEGMENT** key to change the segment parameters.
Err 66	**Module upper temperature limit exceeded. Run terminated.**
Problem:	The module sample temperature exceeded the module's upper limit.
Solution:	Do not try to program temperatures that exceed the recommended upper limit for the module. Use Method Editor to change the final temperatures.
Err 80	**Data input buffer overrun. Data point lost. Software error.**
Problem:	This is a nonfatal error and is not user caused.
Solution:	Call Du Pont for service.
Err 81	**Bad temperature reading. Hardware error. Method terminated.**
Problem:	A problem exists in the hardware or software.

Solution: Call Du Pont for service.

Err 82 **Phase angle not calibrated. Recalibrate for accurate modulus values.**

Problem: The phase angle calibration data has been lost.

Solution: Use the DMA calibration program, or the **INST CALIB** key to recalibrate the phase angle.

Err 83 **Arm zero offset to large. Check arms (locking pins) and sample.**

Problem: When the instrument tried to start the motor, it was unable to bring the arm position to zero.

Solution: Check to make sure the locking pins have been removed, the arms rotate freely, and the sample is mounted correctly. Bring the arm position closer to zero by using the Length Adjust knob.

Err 84 **Cannot start oscillation. Check arm and sample alignment.**

Problem: The instrument could not maintain sample oscillation.

Solution: If in the fixed frequency mode, run the experiment below the resonant frequency range. For high loss samples, press the **START** key or the **MOTOR ON/OFF** key and try again. Try running the sample again at a higher oscillation.

Err 85 **Cannot change mode or number of runs while method or motor active.**

Problem: The error code explains the error exactly.

Solution: Wait until the method or run is completed, or hit the **STANDBY** key before trying to change the mode or number of runs.

Err 86 **Cannot reset parameters while method or motor active.**

Problem: The error code explains the error exactly.

Solution: Wait until the method or run is completed, or hit the **STANDBY** key before trying to reset the instrument parameters.

Err 87 **Run already exists in memory. Cannot start run.**

Problem: You are trying to use a run number that already exists in memory.

Solution: Delete the existing run or change the run number for this run.

Err 88 **Measure at frequency segment not valid in current mode. Cannot start run.**

Problem: You attempted to run a method that has a Measure at Frequency segment, but you are not running in the fixed frequency mode.

Solution: If you want to run an experiment in the fixed frequency mode, change to that mode. Otherwise, select a different method or edit the method to remove the Measure at Frequency segment(s).

Err 89 **Displace segment not valid in current mode. Cannot start run.**

Problem: You attempted to run a method that has a displace segment but you are not running in the stress relaxation or creep modes.

Solution: If you want to run a stress relaxation or creep experiment, change to that mode. Otherwise, select a different method or edit the method to remove the Displace segment.

Err 90 **Phase calibration error. Measured phase exceeds specification.**

Problem: During the phase calibration procedure, the measured phase angle exceeded the instruments specifications.

Solution: Call Du Pont for service.

NOTE

The calibration procedure will continue to run but subsequent data may be inaccurate.

Err 91 **Run data storage area full. Module data storage terminated.**

Problem: You have used up memory allocated for storing data for current run.

Solution: If an important section of the experiment has not yet occurred, start a new run that will cover the experiment, or rerun the experiment using a larger data factor or a larger threshold to reduce the number of points stored.

Err 92 **Module data storage full. Real time disk storage continuing.**

Problem: You have run out of storage area in the module. The experiment will continue, with storage in real time continuing to the 9900.

Solution: Allow the experiment to continue until complete. Do not return to MS-DOS until the experiment is complete because data is only being stored by a real-time data transfer.

Err 93 **No heater power at method start. Check heater position, switch and fuse.**

Problem: The error is exactly as the error explains. The method will start even though power cannot be applied to the heater.

Solution: 1. Check to make sure the oven is installed.

2. Check to make sure heater switch is on. If it is off, turn it on.

3. Check fuse F2. If it is low, replace it.

4. Call Du Pont for service if none of the steps above work.

NOTE

When running an experiment without the oven installed, this error message will occur.

Err 94 Drive signal unstable.

Problem: During calibration, the drive signal drifts from the acceptable range. This error occurs under two conditions. The drive signal did not stabilize within a specified amount of time or the drive signal was stable but became unstable due to bench vibrations or instrument being knocked. You can accept the drive signal if you believe the signal has stabilized adequately.

Solution: 1. Check for bench vibrations.

2. Check to see that the sample is mounted correctly.

3. Call Du Pont for service if none of the steps above work.

Fatal Error Codes

The fatal error codes, the error message, and interpretations are summarized below. Static discharge may cause the instrument to experience a fatal error. To restart the system after a static discharge, reset the system. If the problem persists, contact Du Pont service for further assistance.

Err F00 **Invalid system state detected. Software or memory fault. System halt.**

Problem: A problem exists with the hardware or software and is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F02 **Power Fail.**

Problem: A possible intermittent fluctuation in the instrument's main power or internal problems with the instrument.

Solution: See the discussion on power failure in Chapter 5. If Err F02 persists, call Du Pont for service.

Err F06 **Invalid instruction. Software or memory fault. System halt.**

Problem: A problem exists in the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F07 **CPU watchdog time-out. Software or memory fault. System halt.**

Problem: A problem exists with the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F08 **Stack underflow. Software or memory fault. System halt.**

Problem: A problem exists with the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F09 **Stack overflow. Software or memory fault. System halt.**

Problem: A problem exists with the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F10 **Invalid interrupt request. Software or memory fault. System halt.**

Problem: A problem exists in the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F11 **Unrecognized INT 3 interrupt. Hardware error. System halt.**

Problem: A problem exists with the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F12 **Invalid software state. Software or memory fault. System halt.**

Problem: A problem exists with hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Err F13 **Illegal math function. Software or memory fault. System halt.**

Problem: A problem exists with the hardware or software that is not user caused.

Solution: Call Du Pont for service and then reset the system after repair.

Options

The accessories for the 983 DMA system are briefly described in this appendix. For more information, please contact your nearest Du Pont sales representative. A list of the Du Pont office addresses and phone numbers appears at the end of this section.

Keyboard

The local keyboard is helpful to laboratories which utilize both the 9900 and the 983 systems. The keyboard may be used as a remote experiment controller, freeing the 9900 computer keyboard for data analysis or other applications.

9900 System

A microprocessor based thermal analyzer that controls experiment set-up, thermal programming, data collection, and plotting. The system consists of a Du Pont professional computer, a GPIB interface, an analysis module (and option module interface), and digital plotter.

Liquid Nitrogen Cooling Accessory

The Liquid Nitrogen Cooling Accessory controls the cooling in thermal experiments when used with the 983 DMA system. This accessory can be used with both the Differential Scanning Calorimeter (DSC) and the Dynamic Mechanical Analyzer (DMA) systems.

983 DMA Parts List

PARTS OF THE 983 SYSTEM

General

This section lists replaceable parts for the DMA system.

Parts Ordering Information

For replacement parts, contact the office listed below. To ensure that you receive the correct part for your unit, be sure to include the part number, description, instrument type, model number, and serial number.

Du Pont Instruments
McKean Building
Concord Plaza
Wilmington, DE 19898
Phone (302) 772-5500

983 DMA Parts List

Part No.	Description	Qty.
983005.901	DMA Accessory Kit (consisting of)	1
982166.001	Compliance Sample	1
982165.001	Polycarbonate Sample	4
982161.001	ABS Sample	4
205220.019	Fuse, 1A Amp Slo Blo	1
205220.040	Fuse, 10A Amp Slo Blo	1
280037.000	Wrench, Torque	1
280039.001	Wrench, 1/4 Hex Driver 7/16 Bit	1
290258.001	Wrench, Open End 5/16" x 1/4"	1
264226.001	DMA Oven Cork	1
982093.001	Tension Spring Clamp	2
982080.901	Thermal Lag Device	1

Part No.	Description	Qty.
280255.001	Wrench, 1/16" Hex	1
280257.001	Wrench, 3/32" Hex	1
982154.001	Expandable Locking Pin	1
982160.001	Thin Steel Sample	1
982012.901	Vertical Clamping Mounts (requires Jaw Sets)	2
	(consisting of)	
200967.008	Nut, Hex SSt 5-40	8
200969.005	Lockwasher, Split Size 05	8
280223.001	Screw, Machine Cap Sochd Hex 1/17 x .25	4
280253.001	Screw, Machine Cap Sochd Hex 6-40 x 1	2
982057.001	Clamp Rear Support	2
982058.001	Bushing Contact Clamp Scr	2
982066.001	Clamp Shaft	4
982012.902	Vertical Clamp Jaw Set - Plain	4
982012.903	Vertical Clamp Jaw Set - Serrated	4
	(consisting of)	
982060.001	Jaw Clamp Serrated	2
982064.001	Jaw Stationary Serrated	2
982012.904	Vertical Clamp Jaw Set- Notched	4
	(consisting of)	
982059.001	Jaw Clamp	2
982063.001	Jaw Stationary	2
982011.901	Horizontal Accessory Kit Complete	1
	(consisting of)	
982023.901	Assy Clamp Horizontal Balance Arm	1
982032.902	Assy Clamp Horizontal Motor Arm	1

Part No.	Description	Qty.
207702.021	Screw, Machine Cap Sochd, Hex 4-40 x 0.38	8
980228.901	Assy Fabric Glass Sample Cloth	10
202808.019	Clamp Shaft	4
983023.901	Sample Thermocouple and Ceramic Sleeve	1
982067.001	Sample Tray	1
982068.001	Sample Radiation Shield	1
983006.001	DMA Instruction Manual	1
982221.901	T/C Bracket	1
280150.001	Sample Chamber O-ring Seal	1
982021.982	Furnace Assy (w/exchange)	1
980125	Magnetic Pole	1
980188	Magnet Coil	1
980106	Drive Coil	1
206615	Pivot, Flexure	1
980204.901	LVDT	1

Notice

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For immediate technical assistance with current or potential thermal analysis applications, please call your nearest Thermal Analysis

FOR INFORMATION OR TO PLACE AN ORDER, CONTACT:

UNITED STATES
DuPont Company
Instrument Systems
Concord Plaza, Quillen Bldg.
Wilmington, DE 19898
Telephone: (302) 772-5488
Telex: 835419

BELGIUM
DuPont de Nemours
(Belgium) SA
100 Rue de la
Fusee/Raketstraat
B-1130 Brussels (Haren)
Telephone: (02) 772 07 25
Telex: 62 788

FEDERAL REPUBLIC OF GERMANY
DuPont de Nemours
(Deutschland) GmbH
Dieselstrasse 18
D-6350 Bad Nauheim
Telephone: 06032-306 41
Telex: 4 15 547

FRANCE
DuPont de Nemours (France) SA
137, Rue de l'Universite
F-75334 Paris Cedex 07
Telephone: (1) 45.50.6550
Telex: 206 772

ITALY
DuPont de Nemours
Italiana SpA
FP Department
Via A. Volta 16
1-20093 Cologno Monzese MI
Telephone: (02) 25.302.310
Telex: 314 671

NETHERLANDS
DuPont de Nemours
(Nederland) BV
Helftheuvelweg 1
NL-5222 AV
s'Hertogenbosch
Telephone: (073) 206 510
Telex: 50 53 6

UNITED KINGDOM
DuPont (U.K.) Ltd.
Wedgwood Way
Stevenage, Herts
SG1 4QN
Telephone: (0438) 734 015
Telex: 825591

OTHER COUNTRIES
DuPont Company
Instrument Systems
Export Sales Sections
Concord Plaza, Quillen Bldg.
Wilmington, DE 19898
Telephone: (302) 772-5835
TWX: 510 666 2217
DUPINST WILM.
Telex: 835420

Other offices in Mexico City, Caracas, Sao Paulo. Agents throughout the world.

FOR TECHNICAL ASSISTANCE OR SERVICE IN THE UNITED STATES

HOTLINE

For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis HotLine at (302) 772-5134.

SERVICE

For instrument service and repairs, please call (302) 772-5576.

Glossary

80186	The CPU used in the 983 system.
Address	A number assigned to a device uniquely identifying it as being different from similar devices.
Alphanumeric	A number or letter.
Amorphous	A term generally used to describe polymers totally lacking in crystallinity.
Arm Locking Pins	Those pins used to immobilize the arms and protect the flexure pivots when a sample is being loaded or removed from the DMA.
ASCII	American Standard Code for Information Interchange. A standard 8-bit code used to define a total of 128 characters and control commands.
Assembly Language	A programming language that is machine specific, generally translated from one line of code to one machine instruction. A small portion of the 983 is written in the assembly language for the 80186.
Baud	Transmission rate in bits per second.
Binary	A number system in base 2, using only the digits 0 and 1.
Bit	The storage capacity for one binary digit's worth of information. One bit can store a value that has two states which are typically represented as: 1, 0; true, false; on, off; or Yes, No.

Boolean Value	A value that has only two states. In the 983 and 9900 these values are displayed as Yes and No.
Bus	A path over which information from several sources is transferred to any of several destinations.
Byte	A collection of binary digits, usually eight.
Calipers	A measuring instrument with two legs or jaws that can be adjusted to determine thickness, diameter, and distance between surfaces.
Character Code	The numerical value that represents a character. There are 256 possible characters used by the 983 system.
Checksum	A number calculated by summing all the words in a region of memory. The new sum is checked against the previously calculated checksum to verify that no values have changed since the previous summation.
Clamping Distance	The distance from the axis of the arm to the inside of the clamp face. Used in modulus calculations, the distance between arm centers is the sample length plus twice the clamp distance. Typically: 8.0 $\pm$ 0.2mm.
Cold	An indication that full power is being applied to the heater, but the temperature is still below the set point temperature. The system cannot apply heat fast enough to follow the temperature program.

Compliance — The ability to yield or bend under stresses within the elastic limit. It is the amount of displacement per unit of applied force.

Confidence Test — A test that runs whenever the instrument is first turned on or reset, primarily used to check the electronics in the module base. See Chapter 5.

C Prime (C') — See Drive Signal Constant.

CPU — Central Processing Unit: The hardware that executes the stored program.

Creep Mode — A process, whereby the sample is displaced, and the force is held constant. The sample flexure is monitored as a function of time. The sample is then released to an unstressed state and the sample recovery is monitored.

Crystallinity — A state of molecular structure referring to a long-range periodic geometric pattern of atomic spacings.

Cursor — A movable pointer on the display screen indicating where the next event will occur. It is used most often to prompt you to enter data. A flashing underline (_) is used on the 9900 system and the keyboard of the 983 DMA.

Damping — The ability of a material to dissipate mechanical energy by converting it into heat.

Data Factor — The number of raw data values that are integrated together (averaged) to produce a data point. The data factor is effective only when a method is running.

Data Factor Segment	A segment which allows the modification of the data factor. See Data Factor.
Data File	The information from a thermal experiment stored in a disk file which contains a parameter block, the number of signals, and data points.
Data Point	The experimental information at one point in time consisting of run time, sample temperature, and the measured signals from the experiment.
Data Storage Segment	A segment which turns data collection on (1) and off (0). In the absence of the data storage segment, data storage is automatically initiated with the first Isothermal, Ramp, Measure or Displace segment.
Data Threshold	The minimum data difference required between data points before a data point is stored.
Default	An initial value or condition.
Displacement	The initial arm deflection distance used in stress and creep modes.
Displace Segment	A segment used in stress relaxation and creep experiments to actually perform the displacement and recovery measurements.
DMA	A Dynamic Mechanical Analyzer. Measures changes in the viscoelastic properties of materials resulting from changes in temperature or time.
Drive Signal Constant	A conversion factor from drive signal to acceleration. Nominally: 0.015 to 0.026 mm/(mV sec^2).

Elastomer — A material which can be stretched to at least twice its original length (usually at room temperature) and, upon release of the stress, will return to its approximate, original dimensions.

Epoxy — A class of thermosetting polymers which require curing with a suitable linking agent to develop optimum properties.

EPROM — Erasable Programmable Read-Only Memory. Memory that can be erased with special equipment (usually with intense UV light) and rewritten by applying higher than normal voltages. From then on it can only be read. EPROM retains information even when the power is turned off.

Equilibrate Cycles — The number of oscillations that the sample is subjected to at a new frequency in order to reach mechanical equilibrium. Data collection starts after this equilibrium period.

Equilibrate Rate — The maximum rate of sample dimensional change allowed before a stress relaxation or creep sample is considered to be in equilibrium before an experiment can begin.

Equilibrate Segment — A segment that heats/cools and equilibrates the sample at the defined temperature.

Equilibrate Time — The time period allowed in a stress relaxation or creep experiment for the sample to equilibrate before being flexed.

Event	An auxiliary contact closure (switch) on the 983 module used to synchronize control of user hardware with a method. Event 0 is contact open, 1 is contact closed.
Event Segment	A segment that programmatically controls the external event relay through the event jack located on the module's back panel. 1= on (relay closed); 0= off (relay open).
F1 . . . F12 (function) keys	The twelve keys across the top of the 9900 keyboard.
Fault	An indication that a device has not passed its confidence test.
Field	A particular area of the screen where information, such as name or length, is regularly requested.
Fixed Frequency	A process, whereby the sample is flexed between two parallel arms driven by an electromagnetic driver at an amplitude and frequency selected by the operator. This mode measures the phase lag between the driver and the sample, as well as the amount of power needed to maintain the oscillation amplitude.
Flexure Modulus	A modulus measured when the strain is applied in the flexure mode for long, thin samples.
Frequency (of oscillation)	The number of oscillation cycles completed in one second.
Glass Transition	The temperature at which a material loses its glass-like, more rigid properties and becomes more viscous liquid and flexible in nature.

GPIB	General Purpose Interface Bus. The IEEE-488 standard bus used to connect modules to the 9900 system. This standard defines the electrical and mechanical characteristics of the 24-pin connection between devices. It does not cover the content of the data transmitted.
GPIB Remote State	A state when the 983 is being operated from a remote controller (i.e 9900). All of the keys with the exception of the operate keys, **LOCAL** key, and the **HELP** key are disabled. The **LOCAL** key will return the keyboard to the normal (local control) state.
Hex (hexadecimal)	A number system in base 16. The digits from 0 to 9 are the same as in base 10. Letters A to F represent values of 10 to 15. Hex is convenient for programming because two hex digits are stored in one byte of memory.
Hot	An indication that no power is being applied to the heater and full power is being applied to the LNCA, but the temperature is still above the set point temperature. The system cannot remove heat fast enough to follow the temperature program.
Increment Segment	A segment that raises or lowers the temperature in a step, lets the oven equilibrate, and begins the next segment. This segment can be (+) or (-) depending on which way the user chooses to go.
Inertial Moment	A measure of the effectiveness of mass in motion. Typically, the inertial moment of the 983 arms is 2.4 to 2.7 g m^2.

Initial Temperature Segment	A segment that heats/cools and stabilizes the sample at the defined temperature and then waits for the operator to start the experiment. More than one initial temperature segment may be used in a method.
Integer Number	A number without a fraction.
Isothermal Segment	A segment that holds the sample at the current temperature for a defined period of time.
Jump Segment	A segment that changes the set point temperature, and then immediately executes the next segment.
Kbyte	Kilobyte, equal to 1024 (power of 2 that is closest to 1,000) bytes.
Length Adjust Knob	A knob used to move and adjust the position of the undriven arm assembly.
Length Correction (ΔL)	An empirical correction added to the sample length to account for sample motion that penetrates beyond the clamp faces.
LNCA	A Liquid Nitrogen Cooling Accessory. A self-contained Dewar unit connected to the DMA that provides an external source of coolant and cool gas for the quenching and programmed heating at subambient temperatures.
Local State	A state when the 983 is being controlled from the module keyboard.
Log Factor	The time ratio between saved points in stress relaxation and creep measurements. Used to acquire data at a logarithmic rate.

Loss Modulus	The ability of a material to dissipate mechanical energy by converting it into heat. The absorption of mechanical energy is often related to the movements of molecular segments within the material, such as polymer side chains on specific molecular groups with chains.
LVDT	Linear Variable Displacement Transducer, a device used to measure the position of the arm (amount of sample flexure).
Measure at Frequency Segment	A method segment in the fixed frequency mode used to set the frequency and acquire data points. The temperature is held constant during this segment. Data storage is automatically turned ON to collect the data and is turned OFF at the end of the segment. This segment is used for experiments involving multiple frequencies.
Measurement Points	The number of data points acquired during a Measure at Frequency segment.
Melting Point	The temperature at which crystalline regions in the material melt.
Method	A thermal program composed of one or more steps called segments.
Module	An apparatus containing the heater, sample holder, and transducer used in making a thermal analysis experiment.
Modulus	A quantitative measurement of the stiffness or rigidity of a material.
Morphology	Denotes the internal structure of a material.

Motor Drive Segment	A segment that is used to turn the motor on (1) and off (0). The motor is automatically turned on at the beginning of a method unless the first segment in the method is a Motor Drive off command.
Offline	An indication that a module is no longer communicating with the 9900 system.
Online	An indication that a module is communicating with the 9900 system.
Operating System	A collection of programs that permit the user to manipulate files and control devices which expand the use of a computer.
Oscillation	A periodic motion on the particles of an elastic body in alternately opposite directions from the position of equilibrium when the material is disturbed.
Oscillation Amplitude	The peak to peak amplitude of oscillation in the resonant and fixed frequency modes.
Parallel Port	A connection that uses multiple wires to transmit more than one (usually eight) data bit at the same time.
Parallel Stiffness	Instrument calibration constants that are used to correct for the instrument contributions in the modulus of an extremely soft or thin sample. Typically: Store: 0.30 to 0.40 N m Loss: 0.05 to 0.25 N m at 15 to 21 Hz

Parameter Block	A portion of a data file that contains the sample information, run conditions, and signal descriptions.
PL/M	Programming Language for Microcomputers. A programming language based on PL/I (Programming Language number 1) used to write most of the 983 system's software.
Port	A connection used to transfer data between the CPU and an input/output device.
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 (0.5 for rubbery materials and 0.33 for glassy materials).
Prompt	A phrase that indicates that you are to make an entry to the computer.
Radiant Heat Shield	A shield which keeps the sample from being unevenly heated by the infrared radiation emitted by the oven coils.
RAM	Random Access Memory. The storage area available for temporary data retention in the module.

NOTE

The data is lost from RAM if the power is interrupted.

Ramp Segment	A segment that heats/cools the sample at a fixed rate until it reaches a specified final temperature producing a linear curve of temperature versus time.

Real Number	Any whole (integer), rational or irrational number. A real number is usually represented by an integer, decimal point and a fractional part.
Real Time Plot	A plot of data composed of current information as opposed to stored information.
Relaxation	Internal rearrangements of structural elements which ease stress within a material.
Repeat Segment	A segment that repeats a group of one or more segments within a method, whereby the group of segments will be executed (y + 1) times.
Repeat Til Temp Segment	A segment that repeats a group of one or more segments within a method until the set point temperature exceeds the specified temperature.
Resonant Frequency Mode	A process, whereby the sample is flexed between two parallel arms driven by an electromagnetic driver at an amplitude selected by the operator. This mode measures the frequency of oscillation and the amount of electrical energy needed to maintain the oscillation amplitude. Resonant mode works best when used for low loss, low tan delta materials.
ROM	Read-Only Memory. Memory information that can only be read and retains its information even when the power is turned off.

Run — A thermal experiment. Runs can be stored in memory and later plotted. A run consists of sample and mode information and the data from an experiment.

Run Number — An integer (in the range of 1 to 8388607) used by a module to identify an experimental run.

Run Time — The elapsed time since the start of a method.

Sample Tray — A pan placed directly under the sample to prevent the sample from dripping on the heater coils. Used primarily when experimenting with high temperatures.

Scroll — The up or down movement of information on the display.

Segment — An individual instruction used to form a method.

Serial Port — A connection that uses one wire to transmit one data bit at a time.

Series Compliance — The correction terms used for instrument contributions in the modulus of an extemely stiff sample. These terms cover the bending of arms and the distortion of flexure pivots under extreme loads.
Typically:
Store: 0.5 to 1.5 μm/N
Loss: 0.005 to 0.05 μm/N at 80 to 95 Hz.

Set Point Temperature — The desired temperature for the sample. The temperature controller in the 983 strives to keep the sample at the set point temperature.

Shear Distortion	The distortion in a plane when a substance is under shear deformation (1.3 for cylindrical samples and 1.5 for rectangular samples).
Slide Lock	A locking screw used to rigidly hold the undriven arm assembly into a fixed position while the instrument is running.
Stiffness	The resistance to bending under stresses within the elastic limit (opposite of compliance).
Store	Indication that data from the experiment is being collected and saved.
Strain	The deformation from a specified reference state, measured as the ratio of the deformation to the total-value of the dimension in which the change occurs.
Stress	The force per unit area that tends to deform a body.
Stress Relaxation Mode	A process, whereby the sample is flexed by displacing the arm position a specified amount. The amount of power required to maintain the selected position is then monitored as a function of time. Additional monitoring of the sample occurs when the sample is released from its stressed state, and the sample recovery is monitored.

TA	Thermal Analysis: a group of related techniques that measure some property of a substance as a function of temperature while the substance is subjected to a controlled temperature program.
Tan Delta	The ratio of loss modulus to storage modulus (E"/E' or G"/G'). Tan Delta is a useful index of material viscoelasticity since it is a ratio of viscous and elastic moduli.
Temp*	An indication that a module is running in the calibrated temperature mode where measured temperature is corrected according to a previously run standard's temperature.
Temperature Program	A series of temperatures to which a sample will be subjected. The temperature program is part of a method.
Thermal Lag Device	A thermocouple sleeve that corrects the thermal lag due to thick samples (>5mm).
Thermoplastic	Refers to the class of polymers that are fusible by the application of heat.
Thermoset	A class of polymers with a highly cross linked structure, that are not fusible with the application of heat.
Threshold	The signal deviation limits (signal-to-noise ratio) used to control the data compression function.

Transitions	In most dynamic mechanical damping spectra of polymers, the transitions (peaks) which occur are related to different molecular motions within the polymer. As the temperature is increased, the size of the chains or sections of the polymer involved in the motion increases. These peaks are usually designated starting from the highest temperature damping peak in the spectrum as alpha, beta, gamma, etc.
Version Number	A number automatically assigned as an extension to a data filename that is incremented each time an experiment is run under that filename; or a number used to uniquely identify software, which is displayed on the first screen of a program.
Viscoelasticity	A mechanical property of a substance that exhibits both viscous flow and elastic deformation under rheological conditions.

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