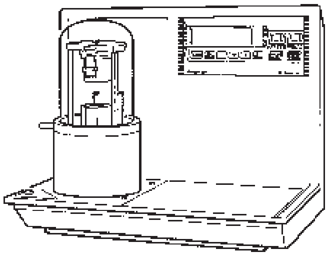
TA Instruments 109 Lukens Drive New Castle, DE 19720

Thermal Analysis & Rheology

A Subsidiary of Waters Corporation



DEA 2970

Dielectric Analyzer

Operator's Manual

PN 925637.001 Rev. B (Text and Binder)
PN 925637.002 Rev. B (Text Only)
Issued September 1997

109 Lukens Drive
New Castle, DE 19720

Notice

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

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions under the guidelines described below:

NOTE:	Highlights important information about equipment or procedures.
♦ ***CAUTION:***	**Emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.**
! *WARNING*	**Marks a procedure that may be hazardous to the operator or to the environment if not followed correctly.**

Helplines

To TA Instruments

For Technical Assistance	(302) 427-4070
To Order Instruments and Supplies	(302) 427-4040
For Service Inquiries	(302) 427-4050
Sales	(302) 427-4000

Safety

This equipment has been designed to comply with the following standards on safety:

- IEC 1010-1/1990 + A1/1992 + A2/1995
- IEC 1010-2-010/1992 + A1/1996
- EN 61010-1/1993 + A2/1995
- EN 61010-2-010/1994
- UL 3101-1, First Edition.

CE Compliance

In order to comply with European Council Directive 89/336/EEC (EMC Directive) and Council Directive 73/23/EEC on safety as amended by Council Directive 93/68/EEC, the following specifications apply to the DEA 2970 instrument:

- *Safety:*
  EN 61010-1/1993 + A2/1995 Installation Category II
  EN 61010-2-010/1994

- *Emissions:*
  EN 55022: 1995, Class B (30–1000 MHz) radiated
  EN 55022: 1995, Class B (0.15–30 MHz) conducted

- *Immunity:*
  EN 50082-1: 1992 Electromagnetic Compatibility—Generic immunity standard Part 1. Residential, commercial, and light industry.
  — IEC 801-2: 1991, 8 kV air discharge.
  — IEC 801-3: 1984, 27–500 MHz, 3V/m.
  — IEC 801-4: 1988 Fast transients common mode 1kV AC power.

Instrument Symbols

The following labels are displayed on the DEA 2970 instrument for your protection:

Symbol	Explanation
	Located on the glass bell jar, drip ring, and top guide shaft plate, ths symbol indicates that the surface may be hot. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.

Please read these labels and take the necessary precautions when dealing with those parts of the instrument. The *DEA 2970 Operator's Manual* contains cautions and warnings that <u>must</u> be followed for your own safety.

Electrical Safety

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

! WARNING

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

! WARNING

After transport or storage in humid conditions, this equipment could fail to meet all the safety requirements of the safety standards indicated. Refer to the NOTE on page 2-9 for the method used to dry out the equipment before use.

Thermal Safety

During an experiment, the furnace, sample, and sensor can become very hot or very cold to the touch.

! WARNING

Allow the sample to return to room temperature before touching the sensor. Take the proper precautions when removing a hot sample.

! WARNING

Do not put your hands inside the furnace. It may be hot enough to cause burns.

Handling Liquid Nitrogen

The DEA 2970 uses liquid nitrogen, as a source of cold gas, in the Liquid Nitrogen Cooling Accessory (LNCA). Because of its low temperature (-196°C), liquid nitrogen will burn the skin. Use extreme caution when working with liquid nitrogen and other cryogenic materials to ensure that you do not burn yourself.

Personnel working with liquid nitrogen should take the following precautions.

! WARNING

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

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

2. Transfer the liquid slowly to prevent thermal shock.

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

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

WARNING

Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent LNCA container in confined spaces.

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

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

IF BURNED BY LIQUID NITROGEN...

1. Flood the area (skin or eyes) with large quantities of cool water IMMEDIATELY; then apply cold compresses.
2. See a doctor IMMEDIATELY if the skin is blistered or if there is a chance of eye infection.

Mechanical Safety

! WARNING

As the ram closes, it generates a signficant amount of force. Make sure you keep your hands and other objects away from this area when the ram is closing.

Lifting the Instrument

The DEA 2970 is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:

! WARNING

Close the furnace before moving the instrument, even for a short distance. Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

Sample Decomposition

The DEA 2970 is capable of heating samples to 500°C. Many materials may decompose during the heating, which can generate hazardous byproducts.

Samples should not be heated above their decomposition temperatures to prevent the release of hazardous materials or contamination of the DEA 2970.

!WARNING

If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

Using This Manual

CHAPTER 1	Introduces the DEA 2970 and lists its specifications.
CHAPTER 2	Describes how to install and assemble your DEA 2970.
CHAPTER 3	Explains the steps needed to set up the sensors and run experiments on the DEA 2970.
CHAPTER 4	Explains the technical aspects of the DEA and its theory of operation.
CHAPTER 5	Provides maintenance and diagnostic procedures.
Appendix A	Lists TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.
Appendix B	Explains the use of the drip pan press in making the aluminum foil drip pans for DEA experiments.
Index	Contains an alphabetical list of topics and page number references.

Chapter 1:
Introducing the DEA 2970

Introduction

The TA Instruments Dielectric Analyzer* (DEA) 2970 is a dielectric analysis instrument used to measure the electrical properties of a material as a function of time, temperature, and frequency. Dielectric measurements characterize the capacitive nature (ability to store electric charge) and conductive nature (ability to transfer electric charge) of the material. The DEA can be used to characterize molecular relaxations and monitor the flow and cure of resins. This information helps the scientist or engineer identify and correlate chemical structure, processing parameters, and end-use performance.

The DEA 2970 works in conjunction with a TA Instruments controller and associated software to make up a thermal analysis system.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instruments
- Enables you to set up experiments and enter constants
- Stores experimental data from the instrument
- Runs data analysis programs.

* The DEA incorporates technology licensed from Micromet Instruments, Inc., Cambridge, MA

Components

The DEA consists of three major parts: the DEA cabinet, where the system electronics are housed; the DEA ram/furnace assembly; and the interchangeable DEA sensors (see Figure 1.1 on the next page). The following components make up the DEA:

- The DEA *cabinet* houses the electronics, valves, *etc.*

- The *keypad* allows you to control and monitor some of the DEA functions from the instrument.

- The *display* is used to monitor the instrument state and the operating parameters.

- The *ram/furnace assembly* contains the following:

  — Interchangeable rams, which are slotted into the top of the assembly.
  — Stepper-motor to move the ram.
  — Linear variable displacement transducer (LVDT) to measure sample thickness.
  — Force transducer to measure the force applied to the sample.
  — Furnace assembly to control the sample temperature with heaters and liquid nitrogen coolant (when connected to the optional LNCA).

- The *interchangeable DEA sensors* are the key to the DEA system; they provide precise measurement in benchtop analysis of bulk sample properties and sample surface properties.

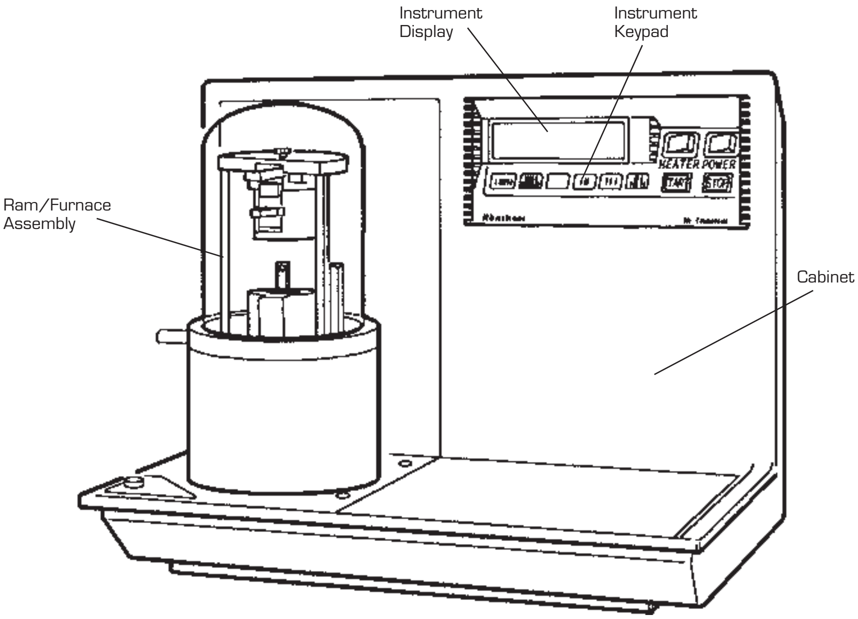

Figure 1.1
The DEA 2970
Instrument

The DEA Instrument

The DEA 2970 instrument contains the electronics and software needed to perform experiments and store the results. The battery backed-up memory in the cabinet saves parameters vital to system operations, if power is interrupted. Also contained in the cabinet is a GPIB interface for communication with the controller.

You can set up, start, stop, and reject the experiment using the keypad on the cabinet or the *Thermal Solutions* Instrument Control software. The display provides valuable realtime information on the experiment in progress.

DEA Display

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

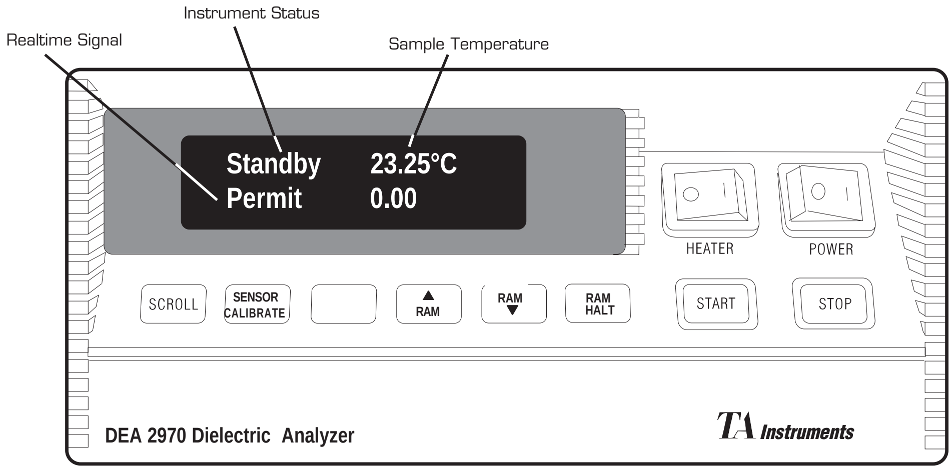

Figure 1.2
DEA 2970
Keypad and Display

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

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

- Instrument status display—the eight-character line on the top left

- Sample temperature display—the nine characters on the top right

- Realtime signal display—the whole bottom line.

DEA Keypad

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

NOTE: Experiment and instrument parameters are entered from the controller keyboard, not the instrument keypad. See the *Thermal Solutions* User Reference Guide.

Table 1.1
DEA 2970
Keypad Functions

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display.
SENSOR CALIBRATE	Closes the ram before a sample is inserted, in order to calibrate the LVDT, zero the force transducer, and make a sensor measurement in dry nitrogen to calibrate sensor response.
NOTE:	The bell jar must be in place for the ram to close.
▲ RAM	Raises the ram to the fully open position to allow you to remove the ram, change the sensors, load a sample, or obtain access to the furnace area.

(table continued)

Table 1.1 DEA 2970 Keypad Functions (continued)

Key/Function	Explanation
RAM ▼	Brings the ram down onto the sample with predetermined force or to a predetermined spacing.
NOTE:	The bell jar must be in place for the ram to close.
RAM HALT	Stops all ram motion, but does not affect the experiment in progress.
START	Initiates the experiment after checking the program method against the mode type. This is the same function as **Start** on the *Thermal Solutions* Instrument Control software.
STOP	If an experiment is running, this key ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions

(table continued)

Table 1.1 DEA 2970 Keypad Functions (continued)

Key/Function	Explanation
STOP *(cont'd)*	go into effect and the data that has been generated is saved. This is the same function as **Stop** on the *Thermal Solutions* Instrument Control software. If an experiment is not running (the instrument is in a standby or method-end state), the STOP key halts any activity (air cool, all mechanical motion, *etc*.).
REJECT (Hold down SCROLL and press STOP)	If an experiment is running, SCROLL-STOP ends the method normally, as though it had run to completion; *i.e.*, the method-end conditions go into effect and the data that has been generated is *discarded*. This is the same function as **Reject** on the controller.

(table continued)

Table 1.1
DEA 2970 Keypad Functions (continued)

Key/Function	Explanation
REJECT *(cont'd)*	SCROLL operates normally (scrolls the text) until the STOP key is pressed. Then the display returns to the signal that was displayed before SCROLL was pressed. If an experiment is not running, SCROLL-STOP halts any activity as described for STOP.

HEATER Switch

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

NOTE: The light in the HEATER switch will glow whenever the power control circuits are enabled. This occurs when either a method is running or an end of method function is selected and active. (See "Heater Indicator Light" in Chapter 5 for more information.)

POWER Switch

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

Accessories

The DEA 2970 can perform experiments on different types of samples using four types of sensors to accommodate a variety of samples and a wide temperature range:

Parallel plate
Ceramic single surface
Remote single surface**
Sputter coated.

The sensors are easily interchangeable. During an experiment, the desired sensor is installed and calibrated; then the sample is loaded onto the sensor. Temperature, frequency, and sample response are transmitted to the electronics in the instrument.

These sensors are shown in the figure below and briefly described on the following pages. For specifications of each sensor type, see Tables 1.1 and 1.2.

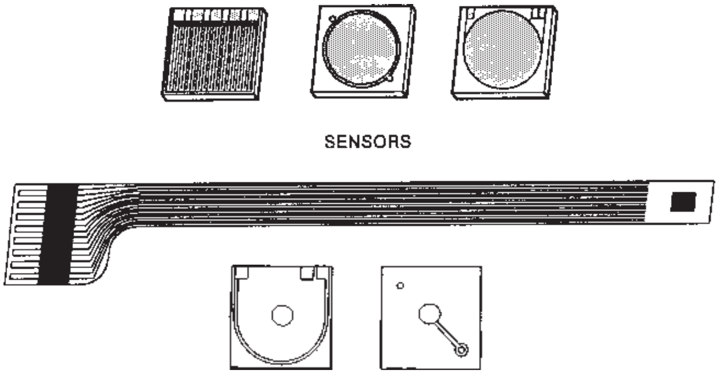

Figure 1.3
DEA Sensors

**The remote single surface sensor is a product of Micromet Instruments, Inc, Cambridge, MA, and is provided to TA Instruments, Inc. for use with the DEA 2970.

Parallel Plate Sensor

Parallel plate measurements are used to evaluate bulk dielectric properties in a material. Traditional parallel plate measurements track molecular relaxations in thick and thin films. The disposable parallel plate sensors and the recessed DEA furnace assembly allow the study of reacting resin systems.

When operating the DEA in the parallel plate mode, the sensors are calibrated by making a capacitance measurement in a dry nitrogen atmosphere. The sample is placed between the two sensor plates after making this capacitance measurement. The stepper-motor then drives the sensors together to a preselected plate spacing or force setting.

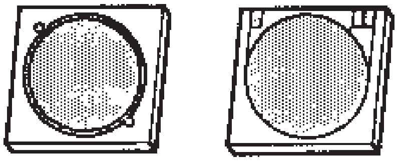

Figure 1.4
Parallel Plate Sensor

Sputter Coated Sensor

Sputter-coated measurements are used to evaluate bulk dielectric properties in a thin film material.

A metallic electrode is sputter coated, under vacuum, directly onto the sample surface to improve sample/measurement electrode contact. To do this, the thin film is mounted inside a mask overlay, which controls the region of sample surface exposed to sputtering. This results in a well-defined electrode (conducting layer) area. Typically, a 300 Angstrom gold layer is then sputtered on each side of the sample.

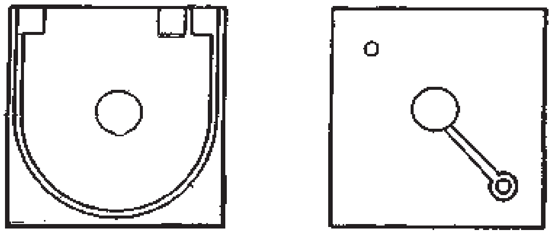

Figure 1.5
Sputter Coated Sensor

Ceramic Single Surface Sensor

The ceramic single surface sensor is used for surface property evaluations and curing experiments, and is ideal for liquid samples.

When operating the DEA in the ceramic single surface mode, the sensor is calibrated by making a capacitance measurement in a dry nitrogen atmosphere. The sample is loaded onto the sensor after making this capacitance measurement. The stepper-motor drives the ram toward the sensor to a preselected thickness or force setting.

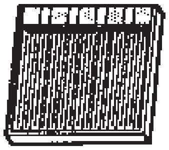

Figure 1.6 Ceramic Single Surface Sensor

NOTE: Dielectric measurements are very sensitive to moisture. Keep the ceramic single surface sensors in a desiccator.

Remote Single Surface Sensor

The remote single surface sensor is a flexible integrated circuit sensor used to evaluate the changes in a material that occur during a cure.

When operating the DEA in the remote single surface mode, you embed the sensor in the sample. The cure then proceeds while the DEA evaluates the changes occurring in the material.

Figure 1.7 Remove Single Surface Sensor

NOTE: Disconnect the remote interface cable from the instrument base when it is not in use.

Specifications

Tables 1.1 through 1.4 contain the technical specifications for the Dielectric Analyzers such as measurement ranges, sampling system, temperature control, and physical specifications.

Table 1.1
DEA Measurement Ranges

Measurement	Range
Frequency range:	0.003 Hz to 100 kHz
Maximum number of frequencies scanned per experiment:	28
Temperature range:	
• Parallel plate sensor	
With LNCA	-150 to 500°C
Without LNCA	Ambient to 500°C
• Sputter coated	
With LNCA	-150 to 500°C
Without LNCA	Ambient to 500°C
• Ceramic single surface sensor	
With LNCA	-150 to 500°C
Without LNCA	Ambient to 500°C
• Remote single surface sensor	-100 to 230°C
	(table continued)

Table 1.1 DEA Measurement Ranges (cont'd)

Measurement	**Range**
Applied voltage:	1 volt
Measured amplitude precision:	0.1%
Phase angle accuracy at 1 kHz:	10^{-4} radians
Tan δ sensitivity at 1 kHz (at 10-sec acquisition rate):	1 x 10^{-4}
Dielectric constant sensitivity at 1 kHz:	0.01
Dielectric constant range:	1 to 10^{5}
Loss factor range:	0 to 10^{8}
Ionic conductivity range:	10^{-5} to 10^{10} pmhos/cm
Force setting range:	0 to 500 N
LVDT precision:	1.0 μm

Table 1.2
Sampling System

Dimensions	Ceramic Parallel Plate	Sputter Coat Parallel Plate	Ceramic Single Surface	Remote Single Surface
Length	25 mm	25 mm	20 mm	3.8 mm
Width	25 mm	25 mm	25 mm	2.5 mm
Minimum Thickness	0.125 mm	0.012 mm	0.125 mm	0.013 mm
Maximum Thickness	0.75 mm	0.75 mm	6.0 mm	no limit

Table 1.3
Temperature Control

Optimum heating rate	0.01°C/min to 5.0°C/min
Cooling rate (with optional LNCA)	
• Down to ambient	0.01°C/min to 10°C/min
• Down to -150°C	1.00°C/min to 5°C/min
Isothermal Stability	± 0.2°C
Oven cool-down time to 50.00°C	30 min

Table 1.4
Physical Specifications

Dimensions	Depth: 17 in. (43.2 cm) Width: 23 in. (58.4 cm) Height: 21 in. (53.3 cm)
Weight	70 lbs (32 kg)
Power	120 Vac 50/60 Hz

CHAPTER 2:
Installing the DEA 2970

Unpacking/Repacking the DEA 2970

You may wish to retain all of the shipping hardware, the plywood, and boxes from the instrument in the event you wish to repack and ship your instrument.

Inspecting the System

When you receive your DEA, look over the instrument and shipping container carefully for signs of shipping damage. Check the parts received against the enclosed shipping list.

If the instrument is damaged, notify the carrier and TA Instruments immediately.

If the instrument is intact but parts are missing, contact TA Instruments.

A list of TA Instruments offices can be found in Appendix A of this manual.

Unpacking the DEA

Refer to Figures 2.1 to 2.3 while unpacking your instrument.

! WARNING

Have an assistant help you unpack this unit. Do not attempt to do this alone.

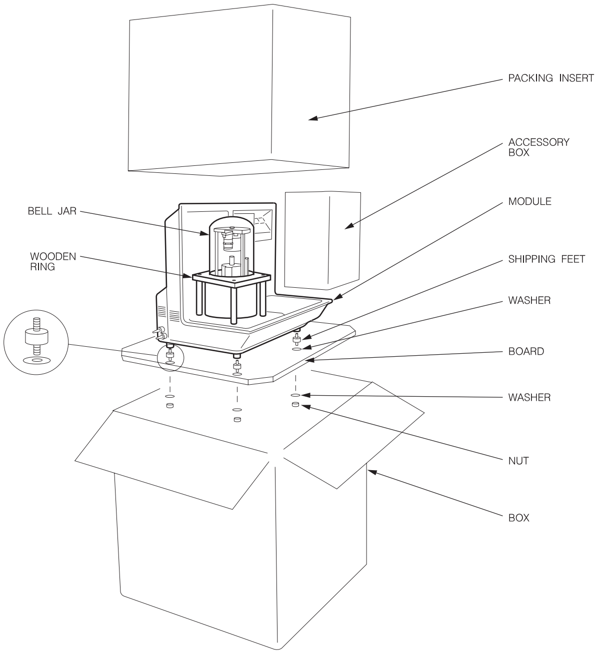

Figure 2.1
Shipping Boxes

1. Open the shipping carton and remove the accessory box.

2. Remove the cardboard packing insert.

3. Stand at one end of the box with your assistant facing you at the other end. Lift your end of the unit out of the box as your assistant lifts his/her end.

4. Place the unit on a lab bench with one side hanging over the edge of the bench (see Figure 2.2). **Someone must be holding onto the unit at all times while it is in this position.**

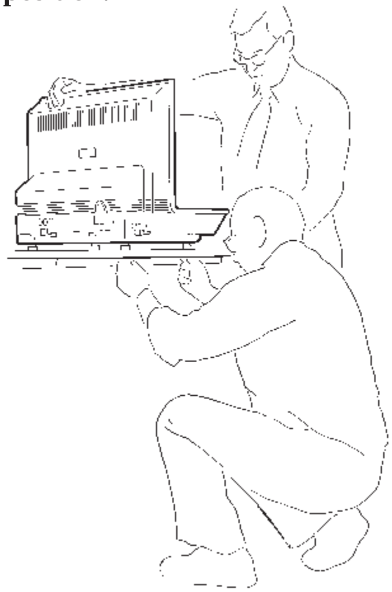

Figure 2.2 Removing the Plywood Board

5. While your assistant holds the unit, use a wrench to remove the nuts and washers from the bottom. Then lift and rotate the unit so that the other end hangs over the edge of the bench. **Someone must hold onto the unit at all times while it is in this position.** While your assistant holds the unit, remove the nuts and washers from the other side. A total of five nuts should be removed.

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

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

8. Remove the mounting feet from the accessory kit.

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

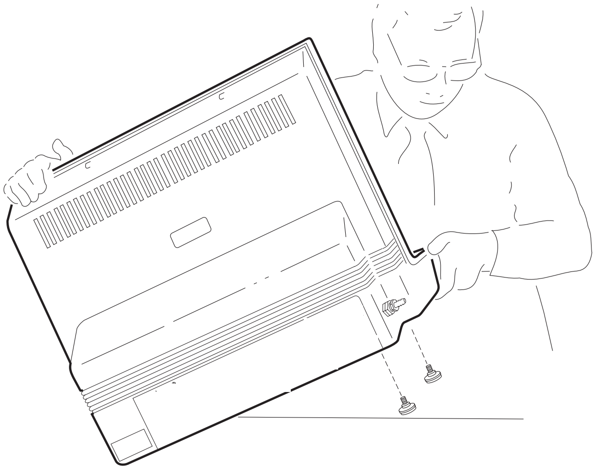

Figure 2.3 Installing the Mounting Feet

Repacking the DEA

To pack and ship your instrument, use the materials retained during unpacking and reverse the instructions found on pages 2-4 to 2-6.

Installing the Instrument

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

- Unpacking the instrument components and accessory kit.
- Inspecting the system for shipping damage and missing parts
- Choosing a location for the DEA
- Connecting the gas lines, accessories, and power cable
- Connecting the DEA to the TA Instruments controller

If you wish to have your instrument installed by a TA Instruments Service Representative, call (302) 427-4000 for an installation appointment when you receive your instrument.

♦ ***CAUTION:*** **To avoid mistakes, read this entire chapter before you begin installation.**

Choosing a Location

Because of the sensitivity of DEA experiments, it is important to choose a location for the instrument using the following guidelines:

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

! WARNING

When using the Liquid Nitrogen Cooling Accessory (LNCA), the DEA exhausts cold vapors, which can cause frostbite, through the furnace outlet port. Position the instrument with the exhaust port pointed away from working areas.

On . . . a stable, vibration-free, heat and fire resistant work surface.

Near . . . a power outlet (120 Vac, 50 or 60 Hz, 10 amps). A step up/down line transformer may be required if the unit is operated from a higher or lower line voltage.
. . . your TA Instruments thermal analysis controller.
. . . sources of compressed lab air and purge gas supply for use during cooling and subambient experiments.

NOTE:

If you are using laboratory purge, rather than bottled purge, you will need to install an external drier.

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

After you have decided on the location for your instrument, refer to the next several sections to unpack and install the DEA.

! WARNING

Drying out the instrument may be needed, if it has been exposed to humid conditions. Certain ceramic materials used in this equipment may absorb moisture, causing leakage currents to exceed those specified in the applicable standards until moisture is eliminated. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following method to dry out the instrument (refer to Chapter 4 for further information):

1 Ramp at 10°C/min to 400°C
2 Isothermal for 30 min.

Connecting Cables and Gas Lines

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

NOTE*: All directional descriptions for this section are written on the assumption that you are facing the back of the instrument.

NOTE: Connect all cables before connecting the power cords to outlets. Tighten the thumbscrews on all computer cables.

♦ CAUTION: **When plugging or unplugging power cords, always handle them by the plugs, not by the cords.**

! WARNING **Protect power and communications cable paths. Do not create tripping hazards by laying them across accessways.**

GPIB Cable

1. Locate the GPIB connector on the right rear of the instrument (see Figure 2.4).

2. Connect the GPIB cable to the GPIB connector. The GPIB cable is the only cable that fits into this connector.

3. Tighten the hold-down screws on the connector.

4. Connect the other end of the GPIB cable to the controller or to the GPIB cable of another TA Instruments instrument connected to the controller.

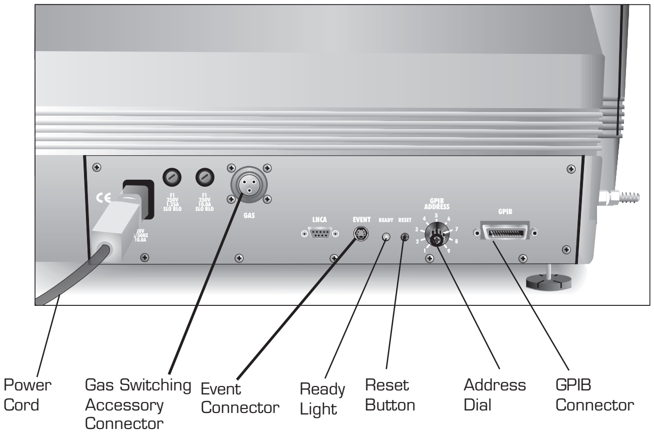

Figure 2.4
DEA Connector Panel

5. Select an address from 1 to 9 (one that is not used by any other instrument connected to your controller). Then use the address selector dial on the DEA connector panel to set the desired address. Figure 2.5 shows a instrument address of 7.

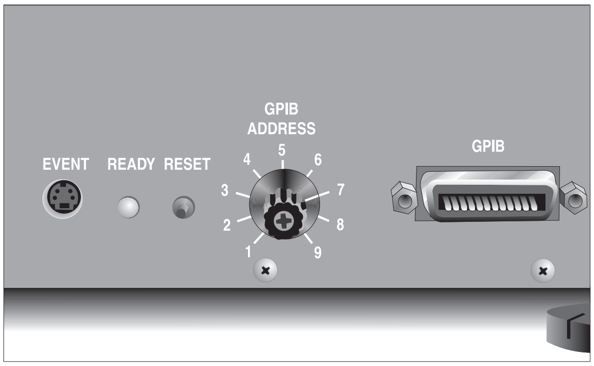

Figure 2.5
Address Selector Dial
(Showing an Address of 7)

NOTE: If you have a multi-instrument system, each instrument must have a different address.

If you change the address after the DEA is powered on, you must press and release the DEA's Reset button to enter the new address. Wait 30 seconds after releasing the Reset button; the green Ready light should begin to glow steadily. Reconfigure the instrument with the controller to bring the instrument back online.

NOTE: The instrument's GPIB address is displayed during startup and can also be viewed on the instrument's status display.

Purge Line

1. Locate the PURGE fittings on the right side of the DEA instrument (see figure below).

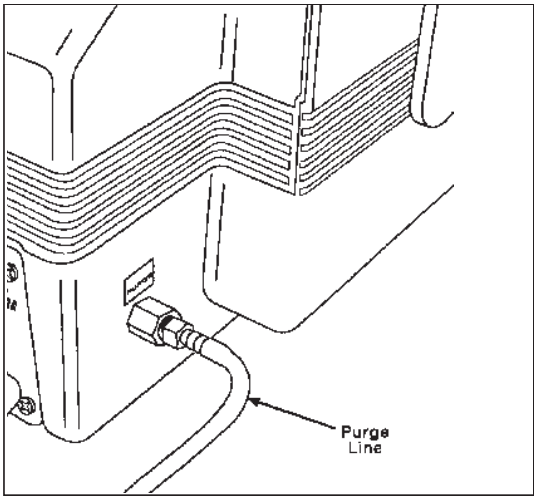

Figure 2.6
Purge Fitting

! WARNING

Use of an explosive gas as a purge gas is dangerous and is not recommended for the DEA instrument.

♦ ***CAUTION:***

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

2. Make sure that the pressure of your purge source is regulated to a maximum of 210 kPa (30 psi).

3. Connect a length of 6.2 mm (1/4-inch) I.D. flexible tubing from the PURGE fitting to the nitrogen source.

NOTE:

If you are using laboratory purge, rather than bottled purge, you will need to install an external drier.

Air Cool Line

1. Locate the COOLING GAS fitting, a 6.2 mm (1/4-inch) compression fitting on the left side of the DEA cabinet back, marked with a 120 psi maximum warning label (see Figure 2.7).

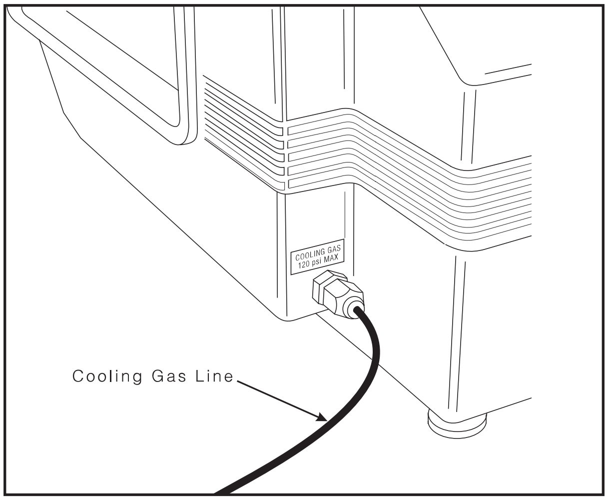

Figure 2.7
DEA COOLING GAS Fitting

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

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

NOTE: You may need to use a filter in the air line to minimize contamination.

Liquid Nitrogen Cooling Accessory (Optional)

To connect the Liquid Nitrogen Cooling Accessory (LNCA) to the DEA 2970, refer to the instructions found in the *LNCA Operator's Manual*.

Power Cable

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

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

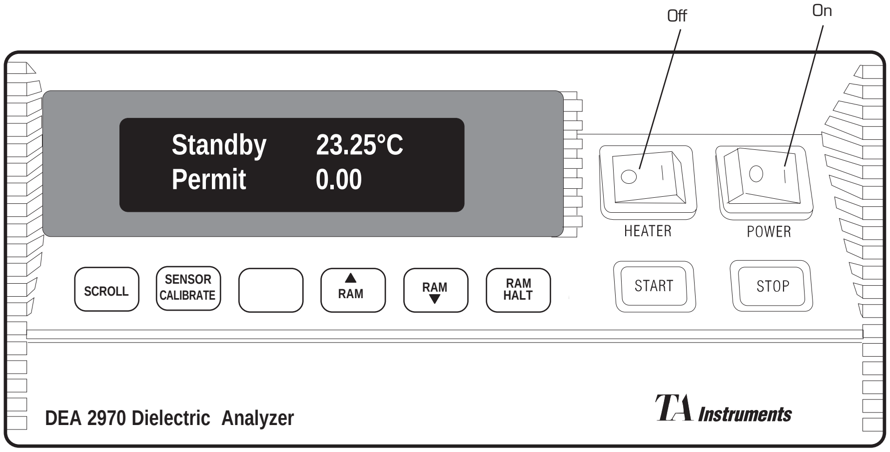

Figure 2.8
Front Panel of DEA
Showing POWER Switch

2. Plug the power cable into the DEA.

♦ ***CAUTION:***

Before plugging the DEA power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.

3. Plug the power cable into the wall outlet.

Starting the DEA

NOTE: Allow the DEA to warm up for at least 60 minutes before performing an experiment.

1. Check all connections between the DEA and the controller. Make sure each component is plugged into the correct connector. (For installation information, see Chapter 2.)

2. Turn the instrument POWER switch to the ON (1) position. The instrument will run an internal confidence test each time you turn on the power.

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

After the confidence test has finished, the screen will briefly display the system status, indicating the amount of data storage memory available and the GPIB address. Next follows the copyright display, and then the standby display.

4. Bring the instrument online with the TA controller.

Shutting Down the DEA

Turning the system and its components on and off frequently is discouraged. When you finish running an experiment on your instrument and wish to use the thermal analysis system for some other task, leave the instrument on; it will not interfere with whatever else you wish to do.

If your system will not be used for longer than five days, we suggest that you turn it off. To power down your instrument for any reason, simply press the POWER and HEATER switches to the OFF (0) position.

Chapter 3: Running Experiments

Introduction

Most DEA experiments follow the same basic set of steps. Use the following instructions as a guide when you perform DEA experiments:

Basic Experimental Steps

1. Select the mode of operation that matches the sensor being used.
2. Choose, set up, and calibrate the sensor appropriate for the sample.
3. Select the instrument parameters that are specific to the mode chosen.
4. Create the method that is appropriate for the sample and selected sensor.
5. Create or select a frequency table to be scanned during the experiment.
6. Mount the properly prepared sample on the DEA.
7. Start the thermal method and perform the experiment.

These steps are explained in the remaining part of this chapter.

Before You Begin

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

- Made all necessary cable connections between the DEA and the controller
- Connected all gas lines
- Powered on each unit
- Installed any desired accessories
- Configured the instrument online with the TA controller
- Become familiar with controller operations
- Calibrated the instrument, if necessary
- Changed the instrument mode, if necessary.

Selecting a DEA Sensor

The DEA can perform experiments on different types of samples using four types of disposable sensors to accommodate a variety of samples and a wide temperature range.

The sensors are easily interchangeable. During an experiment, the desired sensor is installed and calibrated; then the sample is loaded onto the sensor. Temperature, frequency, and sample response are transmitted to the electronics in the instrument. After the experiment has been completed, the sensor set is discarded and not reused.

To determine which sensor to use for a particular experiment, you will need to consider two factors: the sample to be analyzed and the experimental conditions. Refer to the table on the next page to choose the correct sensor for your experiment.

Table 3.1
Sensor Selection

Sample	Experimental Conditions	Sensor
Thermoset		
Liquid or paste	Analysis during cure in controlled thermal history	Ceramic single surface
Liquid or paste	Analysis in prototype mold or external oven	Remote single surface
Cured film	Post-cure analysis	Parallel plate
Thermoplastic *film*	Temperature/frequency analysis	Parallel plate
thin film		Sputter coated
Liquid paint	Analysis during drying or curing in controlled thermal history	Ceramic single surface
	Stability during storage and shipment	Remote single surface
Organic liquid		
Low molecular weight	Ambient temperature measurements	Ceramic single surface or parallel plate
High molecular weight (oil)	Temperature/frequency transition analysis	Ceramic single surface

(table continued)

Table 3.1
Sensor Selection

Sample	Experimental Conditions	Sensor
Sheet molding compound	Maturation/thickening analysis	Remote single surface
	Cure analysis in a controlled thermal history	Ceramic single surface
	Cure analysis in prototype development mold	Remote single surface
Elastomer		
Cured film	Temperature/frequency transition analysis	Parallel plate
Unvulcanized	Analysis during cure	Parallel plate or ceramic single surface

Setting Up the Sensors

Correct sensor installation and sample loading ensure proper operation and accurate results. Before beginning the procedure, you should familiarize yourself with the DEA ram/furnace assembly shown in the figure below.

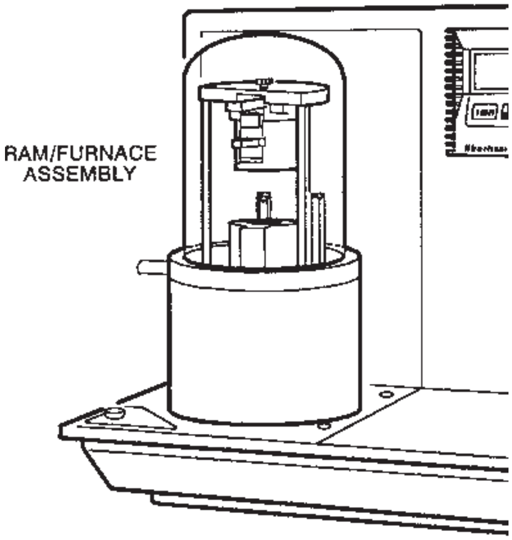

Figure 3.1 DEA Ram/Furnace Assembly

Follow the instructions found on the next several pages to set up the sensors on the DEA.

Parallel Plate Sensor

To set up the parallel plate sensor for analysis, follow these steps:

1. Make sure the instrument temperature is below 50°C. Raise the ram, then remove the bell jar from the ram/furnace assembly.

2. Locate the thumbscrew on top of the ram/ furnace assembly and remove the screw while supporting the ram.

3. Carefully slide the ram toward you until it is separated from the assembly.

4. Place the foil furnace drip pan and sensor into the recess in the furnace, with the sensor contact pads facing to the rear. See the figure below. (Refer to Appendix B for more information on the foil drip pan.)

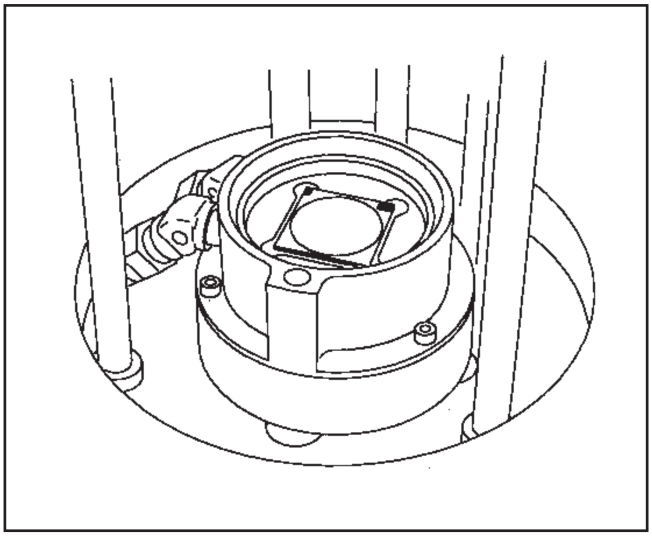

Figure 3.2
Correct Position of
Bottom Sensor

5. Turn the ram upside down. Insert three spring probes into the three holes with gold liners in the ram as shown in the figure below.

NOTE: The spring probes need to be replaced when they are contaminated (by the sample) or have been heated to temperatures over 300°C.

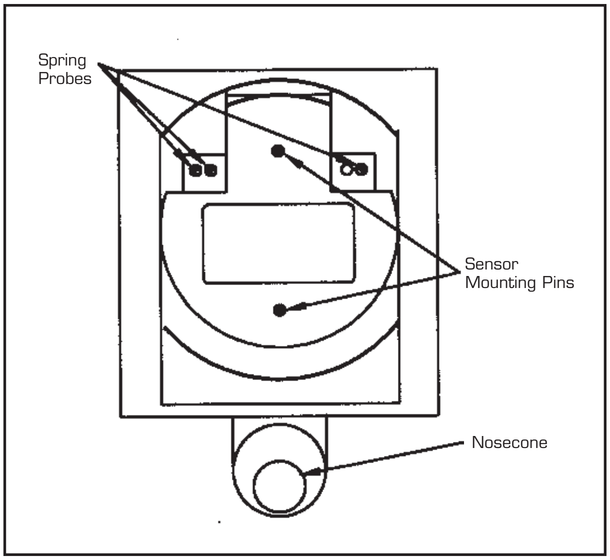

Figure 3.3 Correct Position of Spring Probes in Ram

6. Position the foil ram cover over the parallel plate ram. Make sure the foil cover does not contact the spring probes or sensor mounting pins. Bend the edge of the foil ram cover over the sides of the ram.

7. Press on the sensor release button below the nosepiece on the front of the ram. This will move the mounting pins further apart so that the top sensor plate can be attached to the ram as shown in the figure on the next page.

NOTE:

The sensor release button may be stiff, but it should respond to hand pressure. Do not use tools to move the mounting pins.

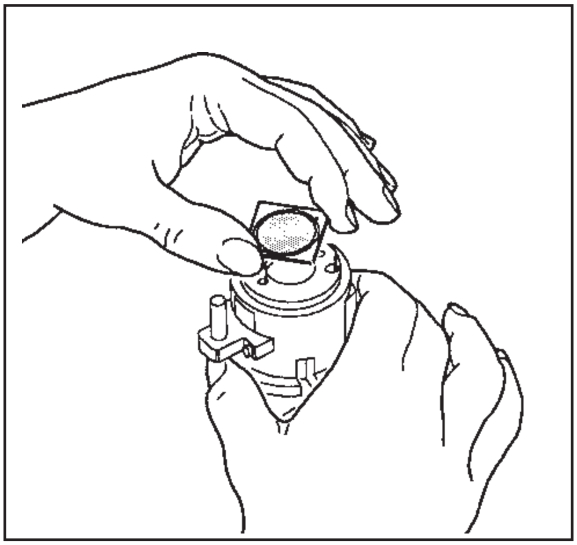

Figure 3.4
Installing the Top Plate of the Parallel Plate Sensor

8. Install the top sensor plate with the guard ring hole mounted toward the rear of the ram. When pressure on the moveable piece is released, the top sensor plate should be firmly attached. See the figure on the next page.

NOTE:

The guard ring hole must be towards the back of the ram.

9. Slide the ram back into the slot in the ram/ furnace assembly until firm contact is made with the back.

10. Supporting the ram with one hand, replace the thumbscrew and turn it down snugly.

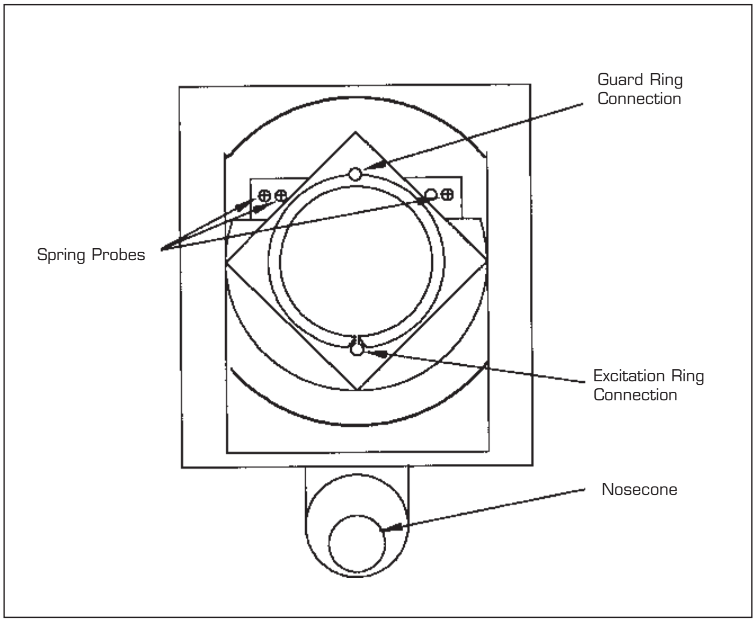

Figure 3.5
Position of Top Parallel Plate Senor in Ram

11. Replace the bell jar and calibrate the sensor (see page 3-22).

Sputter Coated Sensor

To set up the sputter coated sensor for analysis, follow these steps:

1. Make sure the instrument temperature is below 50°C. Raise the ram, then remove the bell jar from the ram/furnace assembly.

2. Locate the thumbscrew on top of the ram/ furnace assembly and remove the screw while supporting the ram.

3. Carefully slide the ram toward you until it is separated from the assembly.

4. Place the foil furnace drip pan and sensor into the recess in the furnace, with the sensor contact pads facing to the rear. See the figure below. (Refer to Appendix B for more information on the foil drip pan.)

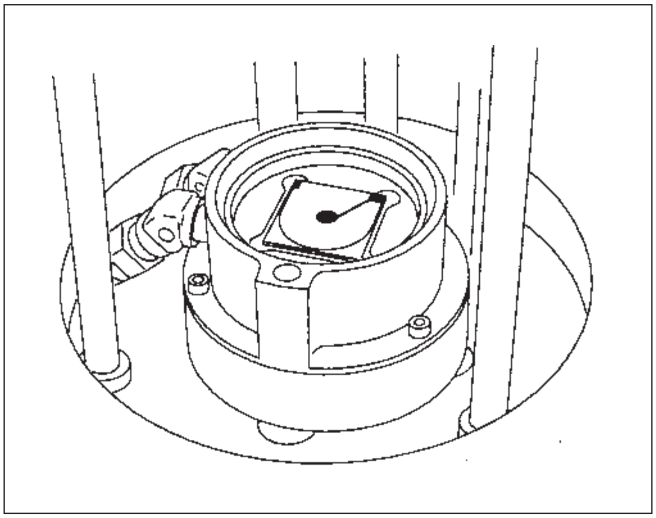

Figure 3.6 Correct Position of the Bottom Sputter Coated Sensor

5. Turn the ram upside down. Insert three spring probes into the three holes with gold liners in the ram as shown in the figure below.

NOTE: The spring probes need to be replaced when they are contaminated (by the sample) or have been heated to temperatures over 300°C.

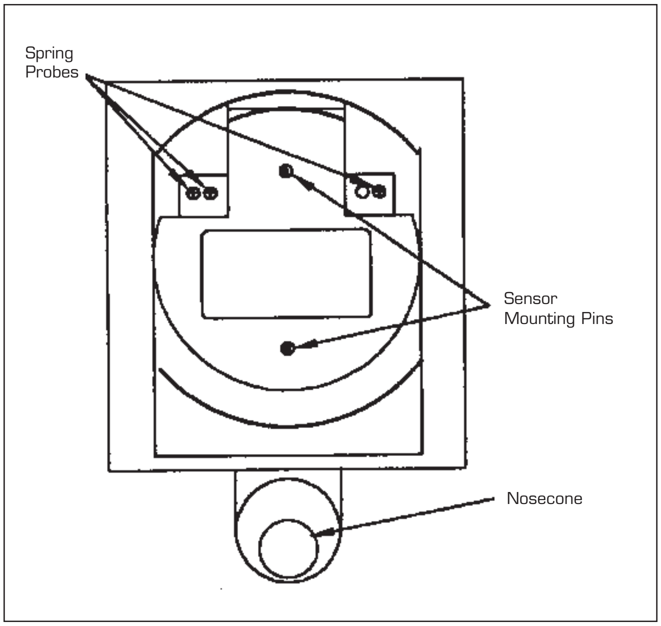

Figure 3.7 Correct Position of Spring Probes in Ram

6. Position the foil ram cover over the parallel plate ram. Make sure the foil cover does not contact the spring probes or sensor mounting pins. Bend the edge of the foil ram cover over the sides of the ram.

7. Press on the sensor release button below the nosepiece on the front of the ram. This will move the mounting pins further apart so that the top sensor plate can be attached to the ram as shown in the figure below.

NOTE: The sensor release button may be stiff, but it should respond to hand pressure. Do not use tools to move the mounting pins.

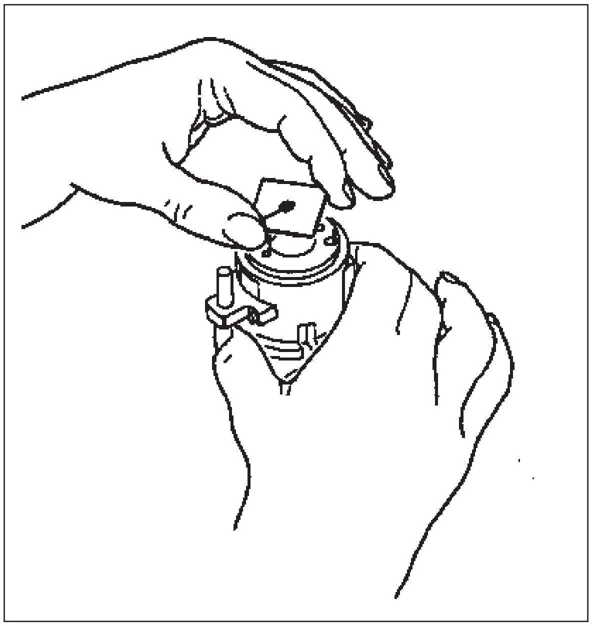

Figure 3.8 Installing the Top Sputter Coated Sensor Plate

8. Install the top sensor plate with the electrode contact hole mounted toward the nosecone. When pressure on the moveable piece is released, the top sensor plate should be firmly attached. See the figure on the next page.

NOTE: The electrode contact hole must be toward the nosecone.

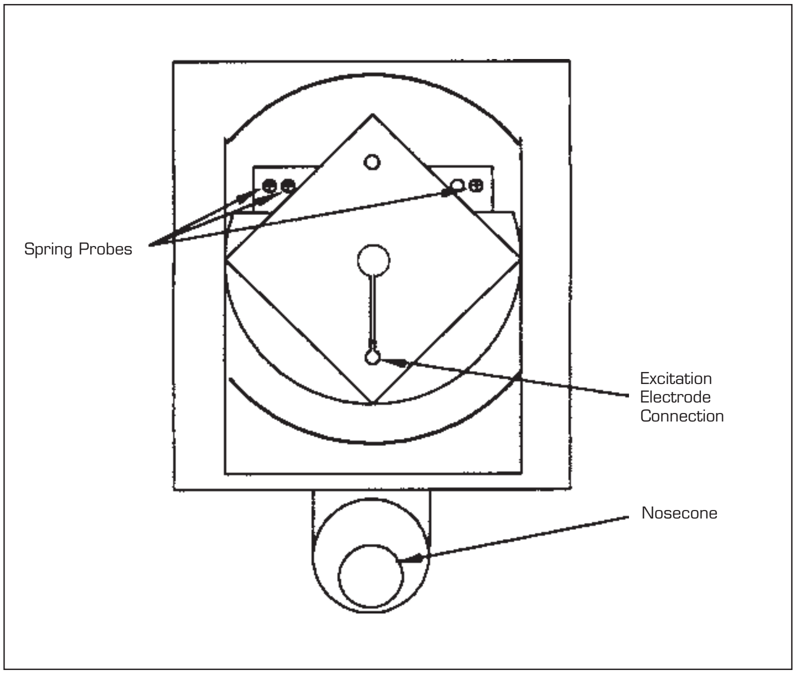

Figure 3.9 Position of Top Sputter Coated Sensor in Ram

9. Slide the ram back into the slot in the ram/furnace assembly until firm contact is made with the back.

10. Supporting the ram with one hand, replace the thumbscrew and turn it down snugly.

11. Replace the bell jar and calibrate the sensor (see page 3-22).

Ceramic Single Surface Sensor

To set up the ceramic single surface sensor for analysis, follow these steps:

1. Make sure the instrument temperature is below 50°C. Raise the ram, then remove the bell jar from the ram/furnace assembly.

2. Locate the thumbscrew on top of the ram/furnace assembly and remove the screw while supporting the ram.

3. Carefully slide the ram toward you until it is separated from the assembly.

4. Place the foil furnace drip pan and sensor into the recess in the furnace, with the sensor contact pads facing to the rear. See the figure below. (Refer to Appendix B for more information on the foil drip pan.)

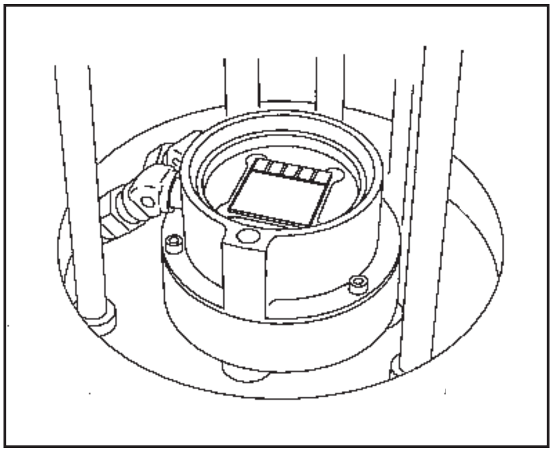

Figure 3.10 Correct Position of Ceramic Sensor in Furnace

5. Turn the ram upside down. Insert five spring probes into the five holes with gold liners in the ram as shown in the figure below.

NOTE: The spring probes need to be replaced when they are contaminated (by the sample) or have been heated to temperatures over 300°C.

6. Position the aluminum ram cover over the ram. Push the ram cover until the edge is seated under the ram cover clips. See the figure below.

7. Slide the ram back into the slot in the ram/ furnace assembly until firm contact is made with the back.

8. Supporting the ram with one hand, replace the thumbscrew and turn it down snugly.

9. Replace the bell jar and calibrate the sensor (see page 3-22).

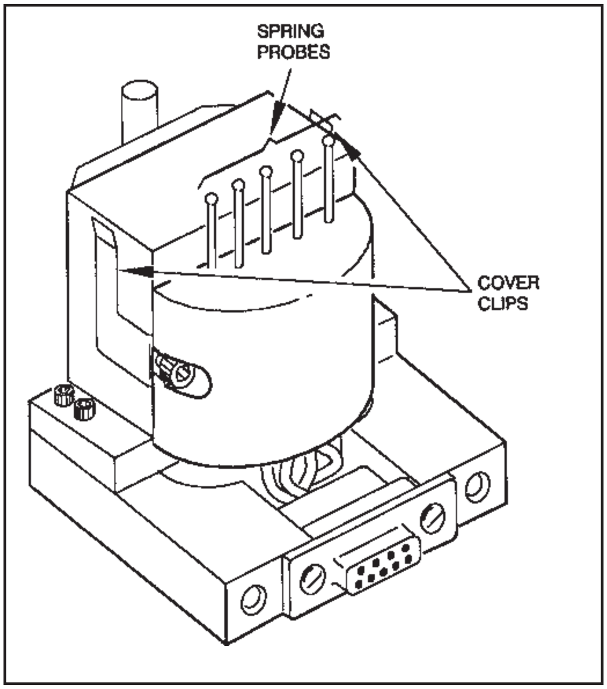

Figure 3.11 Position of Spring Probes and Ram Cover Clips

Remote Single Surface Sensor

To set up the remote single surface sensor for analysis, follow these instructions:

1. Connect one end of the interface cable to the front of the instrument and the other end to the interface box.

2. Insert the connector end of the sensor into the edge connector with the labeled side of the sensor touching the contacts in the edge connector.

3. Insert the edge connector into the interface box with the labeled side up on both pieces.

See the figure below for an example.

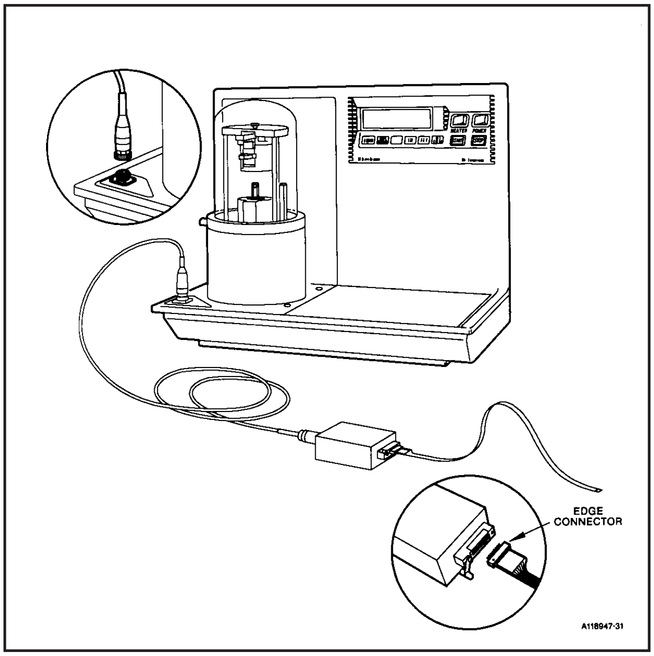

Figure 3.12 Remote Single Surface Connections

After you have connected the remote single surface sensor, you are ready to prepare and mount a sample. See page 3-25 for instructions.

Calibrating the DEA

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

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

Electronic Calibration

Electronic calibration is done to calibrate the DEA analog board. It should be performed at least once a month, and is particularly important with changing weather and laboratory conditions. This is a nine-step procedure that utilizes two calibration fixtures and is done through the *Thermal Solutions* Instrument Control software.

Before you can begin the electronics calibration, perform these preliminary steps:

1. Remove the ram and any sensor(s) from the DEA instrument.

2. Disconnect the remote interface cable from the instrument, if applicable.

3. Install the seven-position DEA internal calibration fixture in place of a ram.

4. Replace the bell jar and purge the enclosure at 0.5 L/min during each step of the calibration.

5. Use the *Thermal Solutions* Instrument Control software to perform the electronic calibration. See the *Thermal Solutions User Reference Guide* for information. (The approximate time needed to perform this calibration is 2.5 hours.)

Sensor Calibration

Sensors are an interchangeable, disposable, part of the DEA instrument. They are the key to the DEA system, providing precise control in benchtop analysis of bulk sample properties and sample surface properties.

The calibration information that you need to either verify or enter will vary depending upon the type of sensor installed.

- To calibrate the remote single surface sensor enter a *Chip ID*, which is a 5- or 6-digit number printed on the connector end of the ribbon cable, as directed in the *Thermal Solutions User Reference Guide*.

- To calibrate the ceramic single surface, parallel plate, and sputter coated sensors follow these instructions:

    a. Install the desired sensor.

    b. Place the bell jar on the instrument.

    c. Turn on the nitrogen purge and allow the DEA to purge for five minutes at a flow rate of 0.5 liters/min. Make sure that the nitrogen purge is complete before you press SENSOR CALIBRATE. Incomplete purging of the atmosphere before sensor calibration can cause the instrument to record low permittivity.

d. Select SENSOR CALIBRATE on the DEA keypad. After approximately one minute, the sensor calibration will be complete and the ram will open.

NOTE: The first time that the SENSOR CALIBRATE key is used, the plunger may not be correctly lined up with the furnace. If this occurs, call TA Instruments for service.

NOTE: Sensor calibration is complete when the ram opens and the instrument status returns to "Standby." Do not stop the calibration procedure before it is complete or your experiment will run without the necessary calibration data.

e. Use the *Thermal Solutions* Instrument Control software (as directed in the *Thermal Solutions User Reference Guide*) to verify/edit the sensor calibration parameters.

These sensors use a *geometry* value, which is the response electrode plate area in mm^2, and an *RTD* value, which is the resistance observed at 0°C for the platinum thermometer on the bottom sensor.

For sputter coated sensors: The geometry used during sensor calibration is the sputter coated area on the sample (mask area). To obtain the value for your particular mask, measure the inside diameter of the mask to +/- 0.01 mm. This value is used to calculate the geometry [$(\pi d^2)/4$]. For the sputter coat mask supplied with this kit, the geometry value is 340 mm^2 +/- 1 mm^2.

Temperature Calibration

Temperature calibration is not usually performed on the DEA, however, it can be done. See the *Thermal Solutions User Reference Guide* for information.

Preparing and Mounting Samples

Sample preparation is an important aspect in achieving accurate and reproducible results. Only one of the sensors require sample preparation *before* they are loaded—only the sputter coated sensor. Turn to page 3-29 for instructions on how to sputter coat and load a sample, then set up the sputter coated sensor.

For the best results, follow these general guidelines for samples:

- Apply a uniform layer of liquid and paste samples to the sensors so that you completely cover the electrode surface. Air bubbles and irregularities will cause erroneous readings.

- Use the fiberglass brush to remove any sample residue from the ram and furnace. Contaminants can cause the sensor to sit unevenly. If the brush does not remove the residue, you may have to heat the ram to 500°C to burn off or soften the residue. Avoid scraping the ram and furnace to remove sample residue.

- Make sure that you calibrate the sensor before loading the sample.

The following pages give you directions on mounting a sample on the various sensors.

Parallel Plate Sensor

For the best results, follow these general guidelines for samples:

- Make sure that hard, solid samples are flat, smooth, and parallel for optimal results.
- When using a liquid sample, use only enough sample to fill the space between the sensors. Avoid flooding the furnace with your sample.
- When using samples that are semi-rigid at room temperature, use a minimum spacing value of one-half the thickness, measured at room temperature.
- If the samples are soft or rubbery at room temperature, use the recommended minimum plate spacing equal to 90 percent of the thickness, measured at room temperature.
- If you encounter erratic behavior in permittivity and loss factor, it might be due to an electric discharge in the sample or to an irregular sample surface. If this occurs, rescan the sample; the second scan should reveal a normal curve.

To load the sample on the parallel plate sensor, follow these steps:

1. Remove the bell jar.
2. Place the sample on the bottom plate using the tweezers provided in the accessory kit.

The sample should completely cover the gold circle, but not the square contact pads as seen in the figure below. You will get an error message if the sample covers the contact pads.

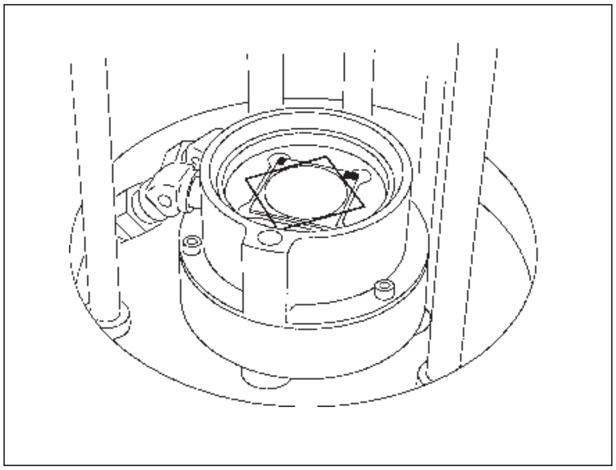

Figure 3.13 Correct Position of Sample on Bottom Parallel Plate Sensor

3. Replace the bell jar.

4. Make sure the nitrogen purge is on.

5. Check to verify that the sample is in place and the heater switch is turned on.

6. Press the RAM ▼ key.

7. Check that the force reading signal is approximately equal to the instrument parameters value selected (+/- 10 N). If the force displayed is more than 20 N below the force selected, lower the minimum spacing value. See the *Thermal Solutions User Reference Guide* for further information.

   See the figure on the next page for the correct assembly of the sample and sensors.

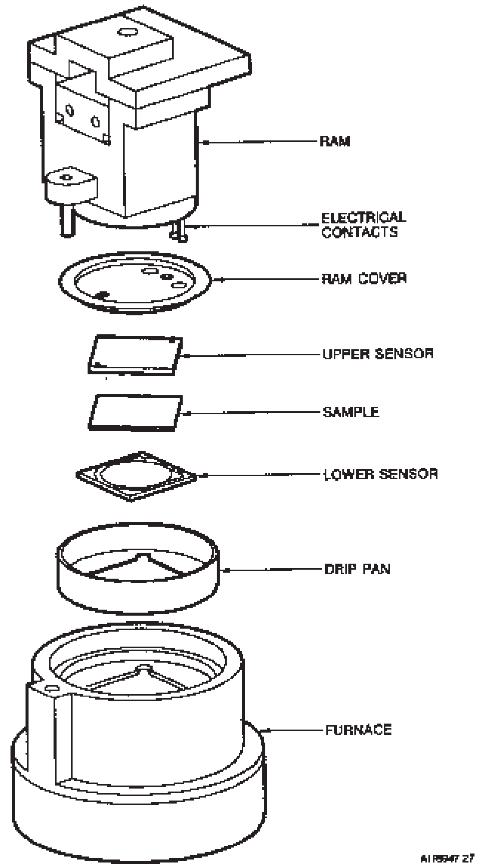

Figure 3.14
Position of Parallel Plate Sensors and Sample

After the sample has been loaded, turn to the section called “Running an Experiment” on page 3-37 for information on setting up and running your experiment.

Sputter Coated Sensor

The sputter coated sensor requires special sample preparation and loading before the sensor can be mounted on the DEA. Follow the directions in this section to prepare a sputter coated sample, then to load that sample and sensor into the DEA instrument.

Preparing the Sample

To prepare a sample for sputter coated analysis you must use a sputter coating device that has a chamber large enough to house the mask. Follow the instructions below to prepare the sample:

1. Cut a 1-inch by 1-inch sample. Measure and record the sample thickness. Measure the inside diameter of the mask to $\pm$ 0.01 mm. This value is used to calculate the geometry. For the sputter coat mask supplied, the geometry value is 340 $mm^2 \pm 1.0$ mm^2.

2. Release the four screws on the sputter coat mask (see the figure on the next page) by turning them 1/4 turn counterclockwise, then remove the top portion.

3. Using tweezers, place the sample on the bottom section, taking care to center the material over the opening.

4. Replace the top section, then tighten the screws by turning them 1/4 turn clockwise.

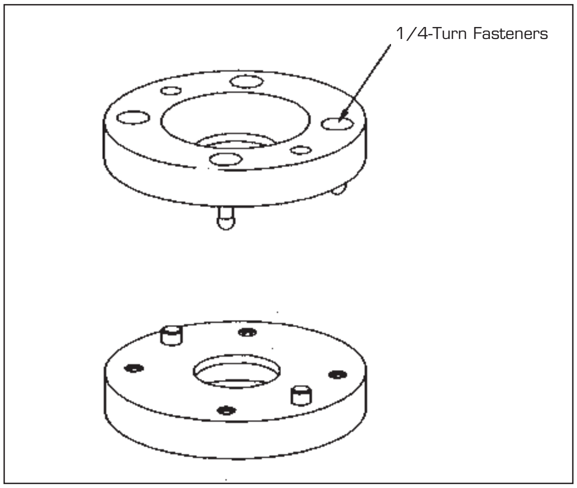

Figure 3.15
Sputter Coated Mask

5. Place the mask in the sputter coater chamber. Sputter coat one side at a time with approximately 300 Angstroms of metallized vapor (usually gold). Follow the manufacturer's instructions when operating the sputter coater device.

6. Remove the mask from the sputter coater chamber and use tweezers to remove the sample carefully.

7. Store the sample in a desiccator until you are ready to load the sample (see the next section) and run your experiment.

! WARNING

ALWAYS use tweezers when you handle the sample. Take care to guard against puncturing the sample electrode.

! WARNING

Materials that are porous may get gold dust into the sample during sputter coating, which can short the electrodes.

Loading the Sample

To load the sample on the sputter coated sensor, follow these steps:

1. Remove the bell jar.

2. Place the sputter coated sample on the bottom plate using the tweezers provided in the accessory kit. Position the sample so that the sputter coated area on the sample is centered on the sensor electrode contact pad without making contact with the RTD ring. The unmetallized portion of the sample should cover as much of the RTD as possible without covering the square contact pads, see the figure below. You will get an error message if the sample covers the contact pads.

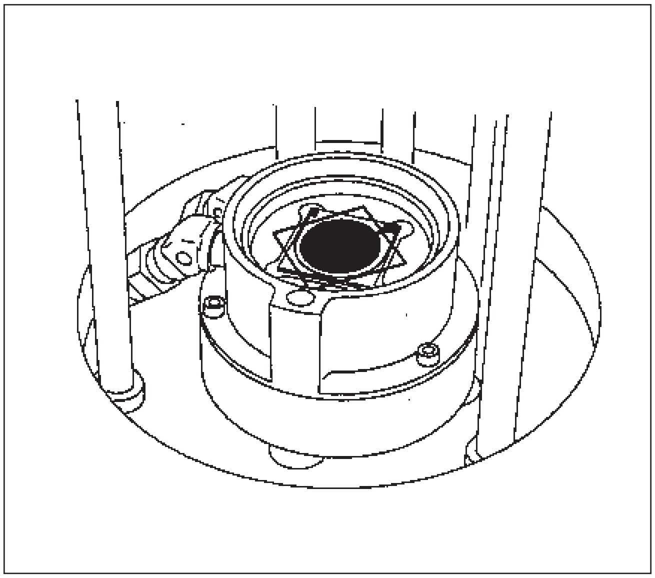

Figure 3.16
Correct Position of Sample on Bottom Sputter Coated Sensor

3. Replace the bell jar.

4. Make sure the nitrogen purge is on.

5. Check to verify that the sample is in place and the heater switch is turned on.

6. Press the RAM ▼ key.

7. Check that the force signal reading is approximately equal to the instrument parameters value selected (+/- 10 N). If the force displayed is more than 20 N below the force selected, lower the minimum spacing value. See the *Thermal Solutions User Reference Guide* for further information.

8. Check that the thickness reading is within 2 microns of total sample thickness. If the thickness is incorrect, enter the correct experimental parameters value.

NOTE: The thickness value entered here, before the experiment starts, is used in the dielectric calculations throughout the experiment.

NOTE: If the LVDT sample thickness disagrees with the external thickness reading by more than 6 microns, remove the sample and recalibrate the sensors.

See the figure on the next page for the correct assembly of the sample and sensors.

After the sample has been loaded, turn to the section called “Running an Experiment” on page 3-37 for information on setting up and running your experiment.

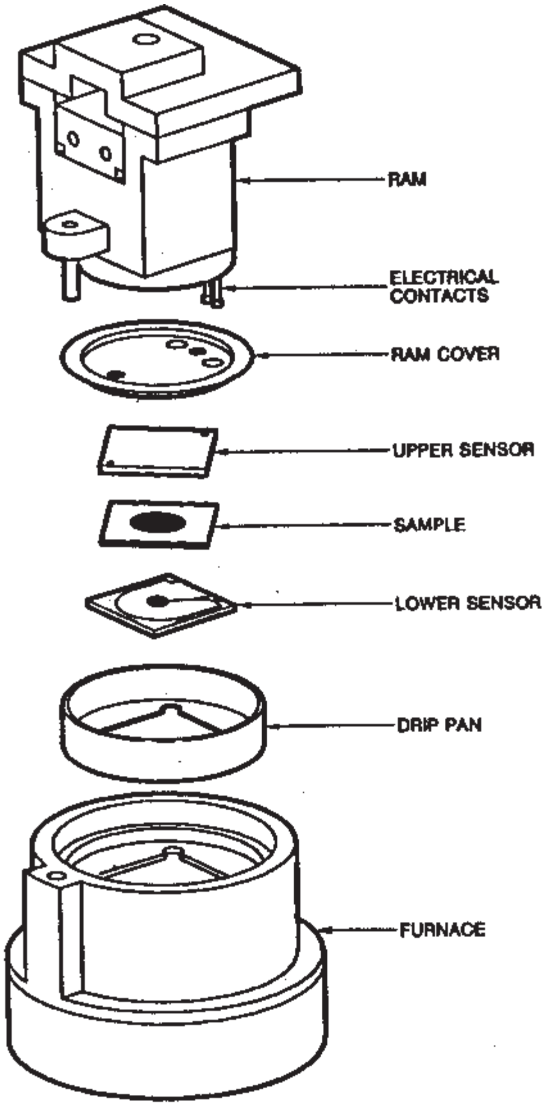

Figure 3.17
Position of Sputter Coated Plate Sensors and Sample

For sputter coated sensors the recommended force is 150 N. Forces less than 100 N may lead to an erratic thickness reading.

Ceramic Single Surface Sensor

WARNING

Make sure that you read the Material Safety Data Sheet (MSDS) before opening the sample packet.

For the best results, follow these general guidelines for samples:

- Make sure that the sample covers the electrode evenly. Things that can cause incomplete coverage are:

  —Contaminants on the furnace floor, causing the sensor to sit unevenly.

  —Resin applied unevenly before it cures.

- Use a minimum spacing of 2.5 mm for liquids and pastes to keep the sample from contacting the ram. This minimizes problems caused by the sample shrinking and swelling.

NOTE: Dielectric measurements are very sensitive to moisture. Keep the ceramic single surface sensors in a desiccator.

To load the sample on the ceramic single surface sensor, follow these steps:

1. Remove the bell jar.

2. Completely cover the entire interdigitated electrode area with the sample, but do not cover the square contact pads as shown in the figure on the next page.

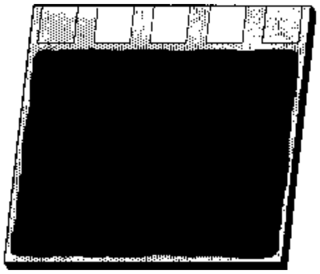

Figure 3.18
Correct Position of Sample on Ceramic Single Surface Sensor

The material must flow between the interdigitated electrodes, which extend 0.013 mm (0.5 mil) above the ceramic substrate. The material must also cover the sensor to a depth of at least 0.125 mm. If the material is viscous at ambient temperatures, the sensor should be removed from the furnace for ease of sample application. You will get an error message if the sample covers the contact pads. After the sample has been applied to the sensor, the sensor can be positioned in the furnace.

3. Replace the bell jar.

4. Make sure the nitrogen purge is on.

5. Verify that the heater switch is on.

6. Press the RAM ▼ key.

After the sample has been loaded, turn to the section called “Running an Experiment” on page 3-37 for information on setting up and running your experiment.

Remote Single Surface Sensor

To load a sample on the remote single surface sensor:

1. Place the end of the remote sensor in a container, if desired, and cover the electrode surface completely with the sample.

2. Make sure the electrode surface is completely wetted with the sample.

After the sample has been loaded, turn to the section called “Running an Experiment” on page 3-37 for information on setting up and running your experiment.

Running a DEA Experiment

Once you have set up the sensors and prepared and mounted the sample, you are ready to perform an experiment.

You will need to set the appropriate instrument and experimental parameters for the DEA using the *Thermal Solutions* software. Refer to the *Thermal Solutions User Reference Guide* for instructions.

When you run experiments on the DEA, follow the instructions given in the next section. The following is a list of general guidelines that may prove helpful when using the DEA 2970 to conduct your experiments.

- Do not reuse any sensors.

- Choose an appropriate heating rate for the experiment from the following equation in order to give at least two data points at a given frequency over a 2.5°C interval:

  Selected
  Heating Rate = 2.5 / Estimated Time* per
  Frequency Scan

  * Found on the *Thermal Solutions* Frequency table.

- *For subambient experiments*: Dry the sample in the nitrogen purge for five minutes before closing the ram and starting the experiment. Increasing the purge rate during the subambient portion of the experiment will minimize condensation.

- *For sputter coated sensors:* The recommended force is 150 N. Forces less than 100 N may lead to an erratic thickness reading.

NOTE: Do not touch the surface of a sensor while setting up an experiment. Oil or dirt on the electrode surface may make a sensor unfit for use.

Starting an Experiment

To start an experiment on the DEA 2970, follow these steps:

1. Use the *Thermal Solutions* DEA Instrument Control program to select the mode, instrument, and experimental parameters.

2. Create and load an experimental method using the *Thermal Solutions* software.

3. Create and load a frequency table using the *Thermal Solutions* software.

! WARNING **Extended operation (periods of time exceeding one hour) above 450°C without adequate purge may reduce the lifetime of the measurement device.**

! WARNING **Do not use the aluminum foil drip pans at temperatures greater than 400°C. Using pans at high temperatures may cause aluminum deposits to form on the furnace.**

4. Set up and calibrate the desired sensor.

! WARNING

Extended operation (periods of time exceeding one hour) above 450°C may cause the sensor to adhere to the furnace. After the furnace cools to ambient temperature, applying dry ice to the top of the sensor normally frees the sensor from the furnace.

5. Prepare and position the sample as directed previously.

6. *All sensors, except the remote single surface sensor*: Use the RAM ▼ key on the instrument keypad to position the ram before the experiment is started. RAM ▼ will produce a measurement at ambient temperatures.

7. Observe the *Thermal Solutions* **Signal Display** window to ensure good starting conditions.

8. Press the START key on the instrument keypad or select **Start** from the menu or tool bar of the *Thermal Solutions* DEA Instrument Control program. The experiment will begin.

Stopping an Experiment

You can stop an experiment before the end of the selected method when necessary. Use one of the following actions to stop an experiment in progress:

- Select **Stop** on the *Thermal Solutions* Instrument Control program or STOP on the DEA keypad to stop an experiment. The "stop" command ends the method, saves the data, then begins the method-end conditions.

- Select **Reject** on the *Thermal Solutions* Instrument Control program or SCROLL-STOP on the DEA keypad to reject an experiment. The "reject" command ends the method normally, as though it had run to completion. The method-end conditions go into effect, but the data that has been generated is discarded.

Removing Samples

When the experiment has run to completion, remove the sample from the DEA as follows:

1. Wait for the instrument to cool to at least 50°C with the ram lowered.

2. *For remote single surface sensor only*: Disconnect the sensor from the interface box, then remove the edge connector from the sensor. Save the edge connector and discard the sensor.

For all other sensors: Press the RAM ▲ key on the instrument keypad. Remove the ram from the assembly. Press on the sensor release button to release pressure from the mounting pins. Remove and discard the sensors and the sample.

NOTE: Do not reuse the foil furnace drip pan or ram cover, if they are deformed or contaminated.

3. Replace the spring probe contacts, if the temperature during the experiment went above 300°C or if the spring probe contacts were embedded in the sample.

Chapter 4: Technical Reference

Introduction

This chapter provides information regarding the theory and applications of the DEA instrument and the dielectric analysis technique.

The following are explained:

- Theory of operation
- Uses for dielectric analysis
- Principles of dielectric analysis
- Quantitative calculations
- Frequency table operation
- Functions of the sensors.

Theory of Operation

Dielectric analysis measures the electrical properties of a material as a function of time, temperature, and frequency. Dielectrics measures two fundamental electrical characteristics of a material:

- *Capacitive* (insulating) nature, which represents a material's ability to store electrical charge.

- *Conductive* nature, which represents a material's ability to transfer electrical charge.

These electric properties are important by themselves, but have even more significance when they are correlated to activity on a molecular level. This correlation allows you to probe the chemistry, rheology (flow), and molecular mobility (relaxations) of polymers and composites.

Uses for Dielectric Analysis

The DEA is used as an investigational tool by various chemical, electronic, pharmaceutical, and manufacturing companies and laboratories. It can also be used in academic work by schools and colleges. The following types of information can be obtained by using the DEA technique:

- resin flow and cure
- surface properties versus bulk properties
- frequency dependence of thermal transitions
- time required to reach full cure
- relative degree of cure
- frequency-dependent transitions at a constant temperature
- influence of cystallinity
- activation energies for transitions
- offline material processing information
- storage stability information
- identification of multiple phases
- change in properties due to exposure to environment (oxidation, thermal breakdown).

Principles of Dielectric Analysis

In dielectric analysis, a sample is placed between two gold electrodes, then a sinusoidal voltage is applied, creating an alternating electric field. This produces polarization in the sample, which oscillates at the same frequency as the electric field, but has a phase angle shift (θ). This phase angle shift is measured by comparing the applied voltage to the measured current (see the figure below).

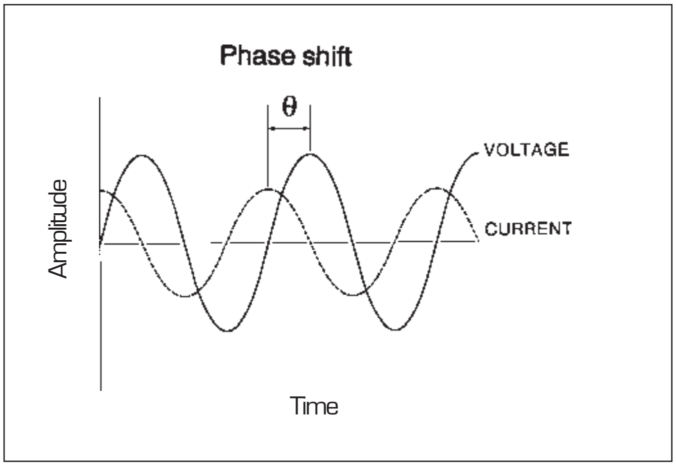

Figure 4.1
DEA Voltage/Current Response

The measured current is separated into capacitive and conductive components via the relationships shown in the figure below.

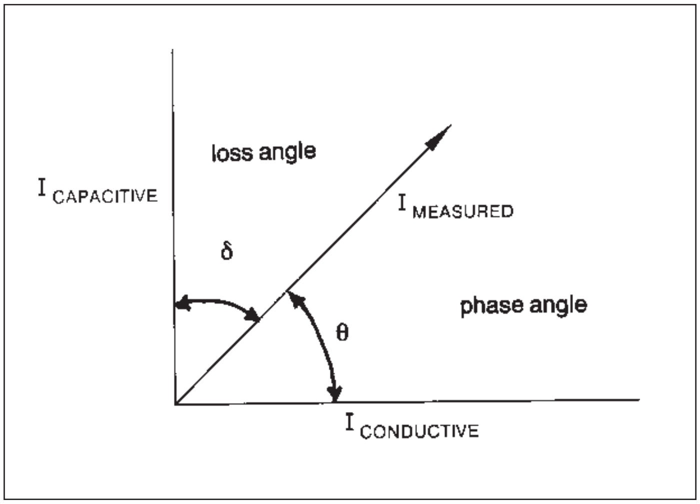

Figure 4.2
Calculation of Capacitive and Conductive Components

Capacitance (C) and conductance ($1/R$) are calculated using the following equations:

$$C \text{ (farads)} = \frac{I_{\text{measured}}}{V_{\text{applied}}} \times \frac{\sin\theta}{2\pi f}$$

$$1/R \text{ (mhos)} = \frac{I_{\text{measured}}}{V_{\text{applied}}} \times \cos\theta$$

where:

R = resistance (ohms)
I = current
V = voltage
f = applied frequency (Hz)
θ = phase angle shift

The current can be measured by a low-impedance current meter or by using the current to charge a load capacitor and then measuring the voltage across the capacitor.

Measurements of capacitance and conductance are used to calculate the following variables, which provide valuable information about molecular motion:

- *Permittivity* (e'), which is proportional to capacitance and measures the alignment of dipoles.

- *Loss factor* (e"), which is proportional to conductance and represents the energy required to align dipoles and move ions.

A *dipole* is a chemical bond that has an unbalanced distribution of charge in a molecule. One part is partially negative and the other partially positive. Permanent dipoles exist in the absence of an applied electrical field, and are caused by the differences in electronegativity of the bonded atoms (*e.g.*, carbonyl bond C=O, C-N). Induced dipoles are those created by the applied electrical field, which causes redistribution of electrons shared between bonded atoms with similar negativity.

For parallel plate electrodes, e' and e" can be calculated as follows:

$$e' = \frac{c\,d}{e_o A}$$

$$e'' = \frac{d}{R\,A\,2\pi f\,e_o}$$

where:

c = capacitance (farads)
R = resistance (ohms) [conductance = 1/R]
A = electrode plate area
d = plate spacing
f = frequency (Hz)
e_o = absolute permittivity of free space (8.85×10^{-12} F/m)

The classic Debye equations for e' and e" are as follows:

$$e' = \underbrace{e_u}_{\text{Permittivity due to induced dipoles}} + \underbrace{\frac{(e_r - e_u)}{1 + (2\,\pi\, f\, \tau)^2}}_{\text{Permittivity due to alignment of dipoles}}$$

$$e'' = \underbrace{\frac{(e_r - e_u)\, 2\,\pi\, f\, \tau}{1 + (2\,\pi\, f\, \tau)^2}}_{\text{Dipole loss factor term}} + \underbrace{\frac{\sigma}{2\pi f\, e_o}}_{\text{Ionic conduction term}}$$

where:

e_u = unrelaxed permittivity
e_r = relaxed permittivity
τ = molecular relaxation time
σ = ionic conductivity

e' represents the amount of alignment of the dipoles to the electric field. e' is low for polymers at low temperature, below thermal transitions, because the molecules are frozen in place and the dipoles cannot move to align themselves with the electrical field. Likewise and for the same reasons, e' is low for highly cross-linked resins.

e" measures the amount of energy required to align dipoles or move ions. Ionic conduction is not significant until the polymer becomes fluid (*e.g.*, above glass transition [Tg] or melting point). Therefore, e" represents the energy required to align dipoles below and through Tg.

Above Tg, e" can be used to calculate the bulk ionic conductivity as follows:

$$\sigma \quad = \quad e'' \, 2\pi f \, e_o$$

Bulk ionic conductivity (σ) can be used to follow the rheological changes that take place during the processing of thermoplastics and the curing of thermosets. Ionic conductivity is related to viscosity, because fluidity is identified by the ease with which ionic impurities can migrate through the sample.

Quantitative Calculations

The TA Instruments DEA provides realtime quantitative calculations of the dielectric parameters, internally converting measured sample response into permittivity and loss factor. These values are also passed to the controller for display and storage. The data file contains:

- e' (permittivity)
- e" (loss factor)
- time
- temperature
- frequency.

In addition, data analysis programs are available with the *Thermal Solutions* library to calculate:

- tan delta
- ionic conductivity
- e* (complex permittivity).

Frequency Table Operation

The DEA instrument is designed to measure sample response at a series of frequencies, enabling you to isolate different kinds of molecular motion and identify frequency dependent molecular relaxations. In addition, the DEA is able to scan isothermally or while ramping the temperature. You specify the frequencies by creating a customized table during set up of the experiment. See the *Thermal Solutions User Reference Guide* for more information on frequency tables.

Function of the Sensors

The TA Instruments DEA has several types of sensors that can be used with the instrument. The basic function of each sensor is briefly described in Chapter 1. A table of sample materials and experimental conditions can be found in Chapter 3 to help you determine which sensor you want to use. This section provides more detail regarding the functioning of the sensors.

Parallel Plate Sensor

The parallel plate sensor is used to evaluate bulk dielectric properties in a material, and to track molecular relaxations. The lower electrode, positioned on the surface of the furnace, applies the voltage that sets up the electrical field and polarizes the sample. A platinum resistance temperature detector (RTD) surrounds the perimeter of the gold electrode and measures the temperature of the sample. The temperature is controlled directly by the RTD. The upper electrode, attached to the face of the ram, measures the generated current, which is then converted to an output voltage and amplified. A guard ring around the perimeter of the upper electrode corrects for electric field fringing and for stray capacitance at the edge of the plates. Signal circuits are connected through pads on the lower sensor, which contact spring probes attached to the ram. The plate spacing (sample thickness) recorded at the start of the method is used throughout the experiment in the calculation of e' and e''.

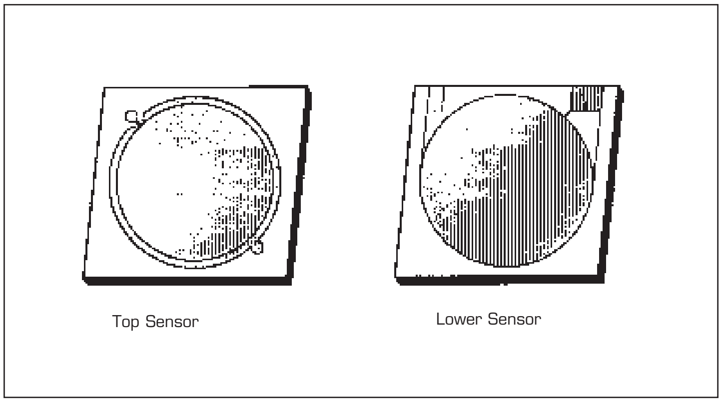

Figure 4.3
Parallel Plate Sensors

Ceramic Single Surface Sensor

The ceramic single surface sensor, based on a coplanar interdigitated-comb electrode design, is used for surface property evaluations and curing experiments. The assembly is composed of a ceramic substrate, metal ground plate, high temperature insulating layer, electrode arrays, platinum resistance temperature detector (RTD), and electrical contact pads. The temperature is controlled directly by the RTD. The sensor is placed on the bottom of the oven and the sample positioned on its top surface. Ram pressure assures intimate sample/electrode contact. Spring probes attached to the ram make contact with pads on the sensor, completing the signal circuits. e' and e" are calculated from the current and phase data using a calibration table stored in the instrument's memory.

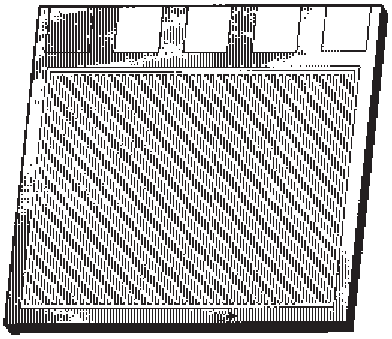

Figure 4.4
Ceramic Single Surface Sensor

Sputter Coat Sensor

Sputter-coated measurements are used to evaluate bulk dielectric properties in a thin film material. A metallic electrode is sputter coated, under vacuum, directly onto the sample surface to improve sample/measurement electrode contact. The lower electrode, positioned on the surface of the furnace, is a contact pad that sets up the electrical field and makes contact with the electrode surface sputtered onto the sample. A platinum resistance temperature detector (RTD) surrounds the perimeter of the gold electrode and measures the temperature of the sample. The temperature is controlled directly by the RTD. The upper electrode, attached to the face of the ram, also acts as a contact pad to make contact with the electrode surface sputtered on the sample. It measures the generated current, which is then converted to an output voltage and amplified. Signal circuits are connected through the pads on the lower sensor, which contact spring probes attached to the ram. The plate spacing (sample thickness) is measured when the ram closes. This can be changed before the experiment is started, and is used throughout the experiment in the calculation of e' and e".

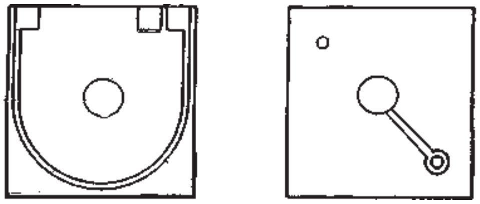

Figure 4.5
Sputter Coat Sensor

Remote Single Surface Sensor

The remote single surface sensor* is used for surface property evaluations and curing experiments. In addition, because of the flexible design and ribbon-cable leads, it can be embedded in a sample of any size for product development. Applications include monitoring dielectric properties of a polymer during molding, or while exposed to adverse environments such as solvents or ultraviolet light. It is also possible to embed the sensor in full-sized prototype products during development for a long-term test of end-use performance or stability and heat history during storage. In this mode, the sample is returned periodically to the instrument for evaluation.

The coplanar interdigitated-comb design of the electrodes is similar to that of the ceramic single surface sensor, but the sensing area is considerably smaller. It uses coplanar, interdigitated-comb electrodes with the electrode array vapor-deposited on a silicon substrate, supported by a carrier of polyamide film and connected to conductors in the ribbon cable. The connector end of the ribbon cable is plugged into an interface box, which is connected to the front of the instrument. The flexibility of the cable and small sensor size, together with the use of a signal amplifier in the integrated circuit adjacent to the sensor array, allows the sensor to

* The remote single surface sensor is a product of Micromet Instruments, Inc., Cambridge, MA and is provided to TA Instruments for use with the DEA 2970.

monitor a sample up to 10 feet away from the instrument. Sample temperature is measured by a thermal diode adjacent to the sensing array. e' and e" are calculated from the current and phase data using a calibration table stored in the instrument memory.

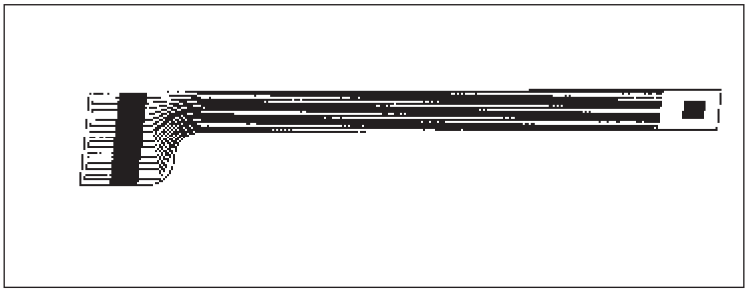

Figure 4.6
Remote Single Surface Sensor

Status Codes

Status codes character strings that are continuously displayed at the top of the controller screen and at the top left of the DEA instrument display. These codes tell you what segment in the method is currently being performed by the instrument.

Table 4.1
Method Status Codes

Code	Meaning
Air Cool	The air cool valve has been turned on to lower the instrument temperature.
Autofill	The LNCA is being refilled from a low-pressure bulk storage tank.
Calib	The method is not running; the instrument is running in a calibration mode, performing sensor calibration or electronic calibration.
Closing	Furnace assembly is closing.

(table continued)

Table 4.1 Method Status Codes (cont'd)

Code	Meaning
Cold	The instrument heater cannot supply heat fast enough to keep up with the thermal program. This may be caused by a large ballistic jump in the program, a faulty heater, or a faulty control thermocouple signal.
Complete	The thermal method has finished.
Cooling	The heater is cooling, as a results of a Ramp segment.
Equilib	The temperature is being equilibrated to the desired set point.
Err *n*	An error has occurred (*n* will appear as a two- or three-digit code). The instrument display will give the error code number and the controller screen also shows the complete error message.
Heating	The heater temperature is increasing, as specified by a Ramp segment.

(table continued)

Table 4.1 (continued)

Code	Meaning
Holding	Thermal experiment conditions are holding; the program is suspended. Press Start to continue the run.
Hot	The instrument is at a higher temperature than the set point, and the instrument cannot remove heat fast enough to follow the thermal program. This is usually caused by a large ballistic jump to a lower temperature or by running a cooling ramp without an LNCA.
Initial	The temperature is being equilibrated to the desired set point. When the temperature has reached equilibrium, the status will change to "Ready."
Iso	The thermal program is holding the current temperature isothermally.
Jumping	The heater is jumping ballistically to the set point temperature.
	(table continued)

Table 4.1 Method Status Codes (cont'd)

Code	Meaning
No Power	No power is being applied to the heater. Check that the heater switch is in the ON (1) position. (See “Heater Indicator Light” in Chapter 5.)
Opening	The furnace assembly is opening.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Press the **Start** key to continue the method.
Reject	The experiment has been terminated and the data erased.
Repeat	The method is executing a repeat loop that does not involve temperature control segments.
Set Up	The system is closing the ram and starting the dielectric measurement prior to starting the first segment of the method.
Stand by	The method is complete.

(table continued)

Table 4.1 Method Status Codes (cont'd)

Code	Meaning
Temp °C	The instrument is in standby mode, and the experiment has been terminated.
Temp *	Temperature calibration is active. The instrument is in standby mode, and the experiment has been terminated, and the motor is not running.

Chapter 5: Maintenance & Diagnostics

Introduction

When you have been using the TA Instruments DEA for a while, some maintenance procedures need to be done in order to keep your instrument running as it should. Several steps are recommended:

- Periodic inspection and cleaning of the instrument.

- Calibration as required—see Chapter 3.

- Replacement of the spring probes, as required—see the instructions later in this chapter.

Inspection

Examine the instrument for good condition as follows:

- Make sure the furnace area is clean and remove any residue, using a fiberglass brush, before starting the next experiment.

- Check the ram for fractures. Surface cracks and discoloration are to be expected.

Cleaning

Cleaning your instrument regularly helps to increase its longevity and maintain its efficiency.

Cleaning the Keypad

You can clean the instrument keypad as often as desired. The keypad is covered with a silk-screen Mylar® overlay that is reasonably water resistant, but not waterproof or resistant to strong solvents or abrasives.

Use a regular household liquid glass cleaner and a paper towel to clean the keypad. Wet the towel—not the keypad with the glass cleaner, then wipe off the keypad and display.

Cleaning the Mounting Pins

The sensor mounting pins may, over time, become encrusted with accumulated sample. Clean the sensor mounting pins periodically with the fiberglass brush provided.

®Mylar is a registered trademark of the DuPont Company.

Disposable Parts

Some of the parts used with the DEA are not meant to be reused more than once, and other parts need to replaced at intervals.

- Sensors are not reusable and should be replaced after every experiment.

- Replace the foil furnace drip pan or ram cover if they become deformed or contaminated with sample.

- Replace the spring probes after any experiment that involves temperatures above 300°C. Spring probes also need to be replaced if they are embedded in the sample when the experiment has been completed.

Replacing the Spring Probes

Check the spring probes after an experiment, if they are embedded in the sample, replace them. If the temperature of the experiment has gone above 300°C, you also need to replace the spring probes. Follow these instructions:

NOTE: You should replace all of the spring probes on a ram at the same time.

1. Use a pair of needle-nosed pliers to pull the probes out of the ram.

2. Place the new probes into their respective openings, using hand pressure to seat them.

Error Messages

As you perform experiments using your DEA, you may see error messages appearing on the display or on the controller.

- If the error is *fatal*, the error message is posted and the system halts.

- If the error is *nonfatal*, the system can continue operating.

The error messages are stored in the error log. To obtain help for an error condition, as the error is displayed on the controller, press the F1 key for online help. To obtain information about the errors contained in the error log, use the *Thermal Solutions* Instrument Control menu to access the log. Online help is available for each error in the error log. See the *Thermal Solutions User Reference Guide* for details.

Diagnosing Power Problems

Fuses

The DEA contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only fuses that you should service yourself are the external fuses, located on the DEA's rear panel. Both are housed in safety-approved fuse carriers, labeled F1 and F2 (see the figure below).

! WARNING

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

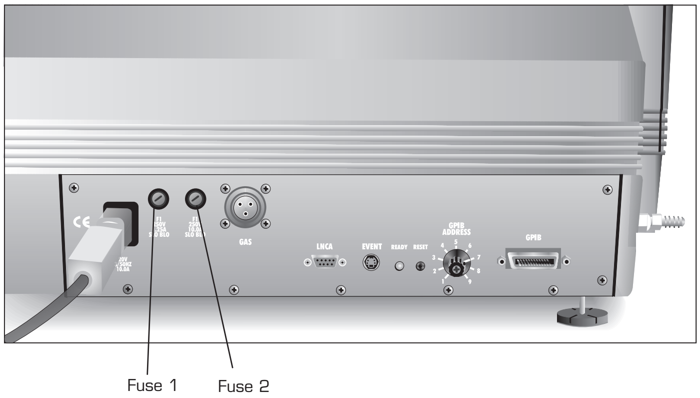

Figure 5.1
Fuse Locations

Fuse 1 is in the circuit between the POWER switch and the instrument control circuits. All power for internal operations and instrument functions, except heater power, passes through this fuse. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Fuse 2 is in the circuit between the main electrical input and the POWER switch.This fuse protects all internal components, including the furnace. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Furnace Power Check

Furnace power is always checked at the beginning of a method. Power supplied to the furnace is switched by a computer-controlled relay as well as by the HEATER switch located on the instruments's front panel. The HEATER switch must be ON (1) to start a method.

NOTE: The light in the HEATER switch will glow only after an experiment is initiated. The HEATER switch will continue to glow, even if it is switched to the OFF (O) position, until the "Temp" status code is displayed.

Heater Indicator Light

The indicator light in the HEATER switch on the front panel of the DEA glows whenever the power control circuitry is enabled. If the light does not come on when the method is started, the indicator light may be defective or a hardware problem may exist in the DEA (call your TA Instruments Service Representative).

The heater light may also remain on after a method has terminated. This can happen if the method-end condition “Return to temperature range” function is chosen; see the *Thermal Solutions User Reference Guide* for further details.

Pressing the instrument **STOP** key after the completion of a method manually overrides the post-experiment heater power conditions.

Power Failures

A power failure caused by a temporary drop in line voltage results in one of two responses by the DEA instrument:

If the drop is fairly large and of long duration (20 milliseconds or more), the system will reset and go into its power-up sequence when power resumes.

If the drop is small or of short duration, the system may halt, and you may see “Err F02” on the display. This message means that the system has detected a power failure and has shut down. The instrument will not restart until it is reset. To reset, press the Reset button on the DEA’s back panel.

If “Err 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 your TA Instruments service representative.

The DEA is designed for a nominal line voltage of 120 Vac (+ 10%), 50 or 60 Hz. It should not be operated outside this range. Low line voltage may result in poor instrument operation; high line voltage may damage the instrument.

DEA 2970
Test Functions

The DEA has three levels of test and diagnostic functions:

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

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

The Confidence Test

The confidence test is run each time the instrument is turned on or reset. The confidence test checks most of the computer and interface components in the system.

When the confidence test is running, the number of the test currently being performed is shown on the display. The test number appears as a two-digit hex number on the lower right of the display. This number is changed as each new test is started. Most of the tests are very brief, so their test numbers may not be apparent.

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

After the tests are completed, a series of sign-on messages are displayed. The system then starts running, and the ready light on the back of the instrument glows.

If an error is detected, an error message is posted on the bottom line of the display. Nonfatal errors are displayed for 3 seconds, and then the confidence test continues. A fatal error occurs when a circuit essential to the operation of the instrument has failed the confidence test; the instrument cannot reliably perform any further functions. The system stops when the fatal error is posted, and the ready light remains off.

Table 5.1, on the next page, summarizes the primary confidence tests for the DEA. If any errors occur during the confidence test, call your TA Instruments service representative.

Table 5.1
DEA Confidence Test

Test Number	Area Being Tested
—	CPU logic
30	CMOS RAM
4*n*	Program memory
5*n*	CPU board I/O functions
6*n*	DRAM data storage memory
70	GPIB test
82	Keypad test
A*n*	Analog board tests
B*n*	Drive board tests
D0	Saved memory checksum

Parts List

The following parts may be ordered for use with the DEA 2970.

Table 5.2
DEA 2970 Parts List

Part Number	Description
205220.021	Fuse-F2 1.25 amp ceramic SLO-BLO
205220.034	Fuse-F1 6 amp ceramic SLO-BLO
265211.001	Bell jar
265303.001	Display
269164.002	Thumbscrew
269364.001	Edge connector
901189.001	Single surface drip cover
901215.001	Drip ring gasket
901220.901	Motor drive printed circuit board
901230.901	Digital signal processor printed circuit board
901240.901	DEA analog printed circuit board
901263.001	Bell jar switch
901264.001	Thermocouple assembly
901265.901	LVDT assembly
901266.901	Stepper motor
901267.901	Heater
901315.901	Parallel plate accessory kit
901316.901	Ceramic single surface accessory kit
	(table continued)

Table 5.2
DEA 2970 Parts List (cont'd)

Part Number	Description
901317.901	Remote single surface accessory kit
901381.901	Remote single surface sensor kit (package of 10)
901382.901	Parallel plate sensor kit (package of 10)
901383.901	Ceramic single surface sensor kit (package of 10)
901385.901	Spring probe kit (package of 30)
901388.901	Foil rounds (package of 20)
901392.901	Foil rounds with holes (package of 20)
990806.901	Air purge valve assembly
890032.001	Keypad
990828.901	Power supply assembly
990850.901	Central processor printed circuit board
984270.901	Communications printed circuit board
990870.901	Triac drive printed circuit board
901402.901	Sputter coated accessory kit
901403.901	Sputter coated sensor kit (package of 10)
901401.901	Sputter coat mask

Appendix A: Ordering Information

For information or to place an order, contact:

United States:

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

Overseas:

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

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

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

TA Instruments Japan
No. 5 Koike Bldg.
1-3-12 Kitashinagawa
Shinagawa-Ku, Tokyo 140
Japan
Telephone: 813/3450-0981
Fax: 813/3450-1322

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

For technical assistance or service in the United States:

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

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

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Appendix B: Drip Pan Press

Introduction

The drip pan press is used to make aluminum foil drip pans for use in DEA experiments. The drip pans prevent any excess sample from running onto the surface of the furnace. See Chapter 3 for information on using the drip pans once they are made. This appendix provides information on the press and how to use it.

To use the drip pan press, you need the following items:

- the drip pan press (see figure below)
- one set of dies to make bottom drip pans
- one set of dies to make top drip pans for the parallel plate ram, with holes to allow the spring probes to seat on the bottom sensor.
- pre-cut foil rounds.

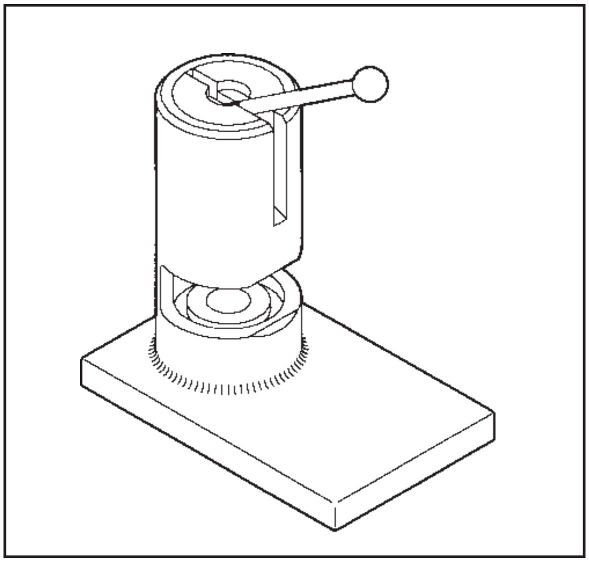

Figure B.1
Drip Pan Press

Using the Drip Pan Press

The foil furnace drip pans are used with the ceramic single surface, parallel plate, and sputter coat sensors. The drip pans are placed into the recess in the furnace as part of the sensor set-up procedures found in Chapter 3.

Setting Up the Press

To set up the drip pan press for use, follow these steps:

1. Place the grooved die in the bottom of the press. The pins in the bottom of the press should line up with the groove to ensure proper alignment of the dies.

2. Place the top die in the top of the press with the keyhole facing the front. Tighten using a 1/16-inch hex key.

Manufacturing the Drip Pans

To make the foil drip pans, follow the steps below:

1. Place a pre-cut foil round in the bottom of the drip pan press.
2. Lower the press handle with a smooth stroke, then release the handle.
3. Gently remove the drip pan from the press and inspect it for tears and flaws.

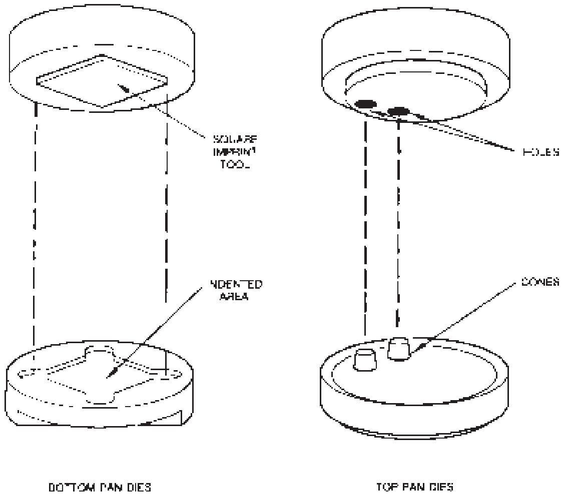

Figure B.2
Drip Pan Dies

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