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DSC 2910

Differential Scanning Calorimeter

Operator's Manual

PN 925600.001 Rev. E (Text and Binder)
PN 925600.002 Rev. E (Text Only)
Issued June 2000

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New Castle, DE 19720

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

!WARNING

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

◆ ***CAUTION:***

A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

NOTE:

A NOTE highlights important information about equipment or procedures.

Safety

Instrument Symbols

The following labels are displayed on the DSC 2910 instrument for your protection:

Symbol	Explanation
	Indicates the presence of one or more of the following: hazardous voltage, high temperature parts, or moving parts.
	Indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DSC 2910 Operator's Manual* contains cautions and warnings that must be followed for your own safety.

Safety

(continued)

Electrical Safety

WARNING

The pushbutton switch next to the cell connector, located on the cell base, provides the AC power interlock for the cell. Do not press down on the switch with anything other than the cell as you install it. This supplies voltage to the cell connector when depressed; there is danger of electric shock should it be depressed accidentally. Turn the HEATER switch to the off (O) position when installing or removing cells to reduce the possibility of an electrical shock.

You must unplug the instrument before doing any maintenance or repair work; voltages exceeding 110 volts AC are present in this system.

WARNING

High voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the cabinet covers. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

An isolation transformer should be used when troubleshooting. *

* Test equipment may connect the instrument to ground, rendering the Isolation Transformer ineffective. There are low voltage circuits in this equipment that are referenced to hazardous voltages.

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 2-8 for the method used to dry out the equipment before use.

Handling Liquid Nitrogen

The DSC 2910 uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature [-195°C (-319°F)], liquid nitrogen will burn the skin. When you work with liquid nitrogen, use 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 displacement of oxygen in the air.

1. Wear goggles or a face shield, gloves 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 tops.

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

...continued on page xvii



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.

Safety

(continued)

3. The purity of liquid nitrogen decreases as the nitrogen evaporates. If much of the liquid in a container has evaporated, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

IF A PERSON IS BURNED BY LIQUID NITROGEN . . .

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

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

Chemical Safety

WARNING

Do not use hydrogen or any other explosive gas with the standard DSC 2910 cells. There is a special version of the DSC 2910 Pressure cell (PN 900830.901) that has been modified to allow hydrogen gas to be used in it.

Hydrogen gas should be used with extreme care. It is highly flammable when exposed to flame or oxidizing materials. When using hydrogen in the pressure DSC cell, the cell should be initially purged thoroughly with helium before introducing hydrogen. At the end of the experiment, the cell should be vented into an exhaust hood and repurged with helium prior to opening the pressure container.

Safety

(continued)

◆ ***CAUTION:*** Use of chlorine gas will damage the cell.

!WARNING **If you are using samples that may emit harmful gases, vent the gases by placing the DSC near an exhaust.**

Thermal Safety

The cell surfaces can be hot enough to burn the skin during a sample run. The exit gas from the furnace tube on the 1600°C DTA cell is extremely hot; keep hands and any combustible material away from this exit gas flow.

If you are conducting a subambient test on the DSC, cold could also cause injury. After running any type of experiment, you must allow the DSC cell and the 1600°C DTA cell to return to room temperature before you touch the inner cell surfaces.

Using This Manual

Chapter 1	Describes the DSC 2910 and its specifications.
Chapter 2	Describes how to connect the DSC 2910 to the rest of your system and install the various cell types.
Chapter 3	Describes how to run DSC, Pressure DSC, and 1600°C DTA experiments.
Chapter 4	Provides technical information, principles of operation for the cells, and guidelines.
Chapter 5	Describes instrument maintenance procedures and the confidence test codes.
Appendix A	Explains how to change the Sample Encapsulating Press dies.
Appendix B	Lists worldwide TA Instruments offices that you can contact to place orders, receive technical assistance, and request service.

Using This Manual

(continued)

Appendix C	Tells you how to use the Modulated DSC® option.
Index	Lists the page numbers of important topics for your reference.

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Introduction

The Differential Scanning Calorimeter (DSC) 2910 determines the temperature and heat flow associated with material transitions as a function of time and temperature. It also provides quantitative and qualitative data on endothermic (heat absorption) and exothermic (heat evolution) processes of materials during physical transitions that are caused by phase changes, melting, oxidation, and other heat-related changes. This information helps the scientist or engineer identify processing and end-use performance.

The DSC 2910 instrument works in conjunction with a 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
- Runs data analysis programs.

Components

The DSC 2910 (see Figure 1.1) has two major parts: the 2910 instrument, which contains the system electronics, and the cell, which contains its own thermocouples (temperature sensor) for monitoring differential heat flow and temperature. Three interchangeable cell types are available:

- Standard DSC Cell
- Pressure DSC Cell
- 1600°C Differential Thermal Analysis (DTA) Cell.

The figure below identifies the parts of the instrument.

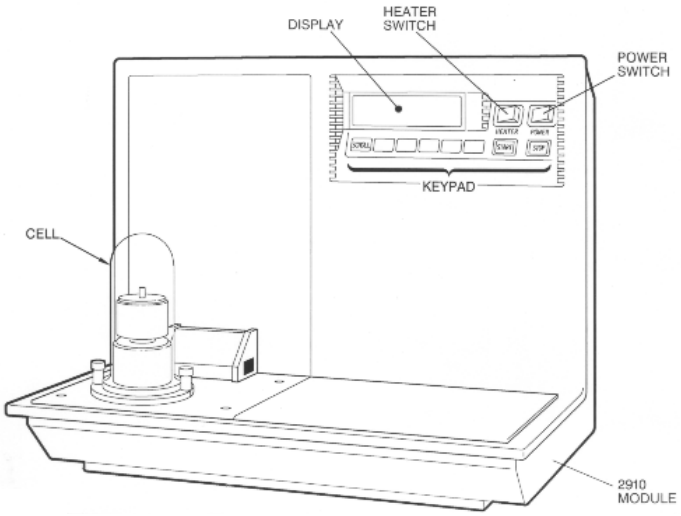


Figure 1.1
DSC 2910

The 2910 Instrument

The DSC 2910 contains the electronics and software needed to perform experiments and store experimental results. The battery backed-up RAM in the instrument saves parameters vital to system operations if power is interrupted. Also contained in the instrument is a GPIB interface for communication with the controller.

The keypad on the front of the DSC 2910 enables you to start and stop experiments. The display above the keypad provides realtime information about the experiment.

The DSC 2910 also contains several hook-ups for other components and accessories in the thermal analysis system, including:

- Gas purge
- Cool-down line
- Vacuum
- LNCA (Liquid Nitrogen Cooling Accessory)
- Gas Switching Accessory
- EVENT relay
- GPIB
- Power cable.

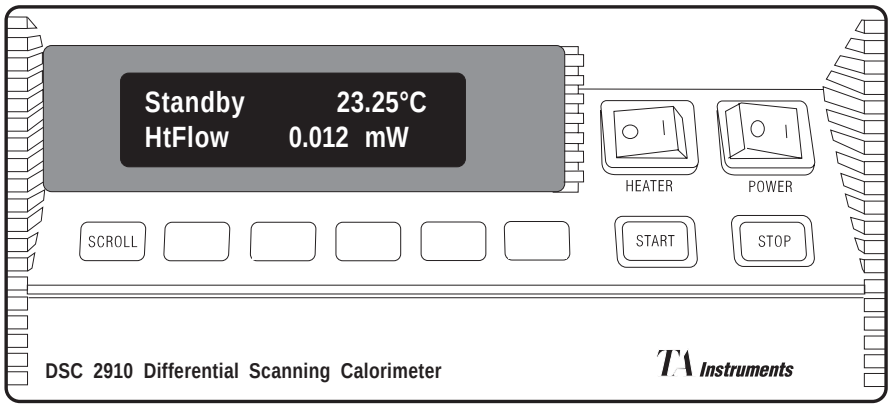


Figure 1.2
DSC 2910
Display and Keypad

2910 Display

The DSC 2910 display is the lighted area above the keypad (see Figure 1.2). It contains two rows of 20 characters each.

During normal operation, the display is segmented into three areas. The left eight characters on the upper line show the instrument status; the right nine characters show the sample temperature; and the bottom line is a realtime signal display. Instrument status codes are described in the Technical Reference chapter of this manual.

2910 Keypad

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

NOTE:

Experiment information and instrument constants are entered from the controller keyboard, not the instrument keypad.

Table 1.1
DSC 2910 Keypad Function Keys

Key/Function	Explanation
SCROLL	Scrolls the realtime signals shown on the bottom line of the display. For more information on the progress of the experiment, refer to the status and signal displays on the controller.
START	Initiates the experiment after the method is checked against the cell type. This is the same function as **Start** on the controller.
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 selected go into effect, and the data that has been generated is *saved*. This is the same function as **Stop** on the controller.

(table continued)

Table 1.1
DSC 2910
Keypad Function Keys
(continued)

	If an experiment is not running (the instrument is in a stand-by or method-end state), STOP halts any activity (air cool, LNCA auto-fill, *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.
NOTE:	The SCROLL key operates normally (scrolls the realtime signals) until the STOP key is pressed. If an experiment is not running, SCROLL-STOP works like the STOP key.

HEATER Switch

The HEATER switch (see Figure 1.2) turns the power to the instrument heater on and off. The switch should be in the ON position before you start an experiment.

NOTE: The heater light will glow whenever power is being supplied to either the heater coils in the furnace or the Liquid Nitrogen Cooling Accessory. The heater light may also remain on after a method has been terminated. (See Heater Indicator Light in Chapter 5 for more information.)

POWER Switch

The POWER switch (see Figure 1.2) turns the power to the DSC 2910 on and off.

Standard DSC Cell

The standard DSC Cell (Figure 1.3) is used to measure differential heat flow. The sample and a reference are placed in pans that sit on raised platforms on a constantan disk, and heat is transferred through the disk up into the sample and reference. The differential heat flow is monitored by thermocouple wires welded to the disk.

NOTE: When using the Refrigerated Cooling System accessory with the 2910, a special RCS-DSC cell must be installed on the instrument. Refer to the RCS addendum for further details.

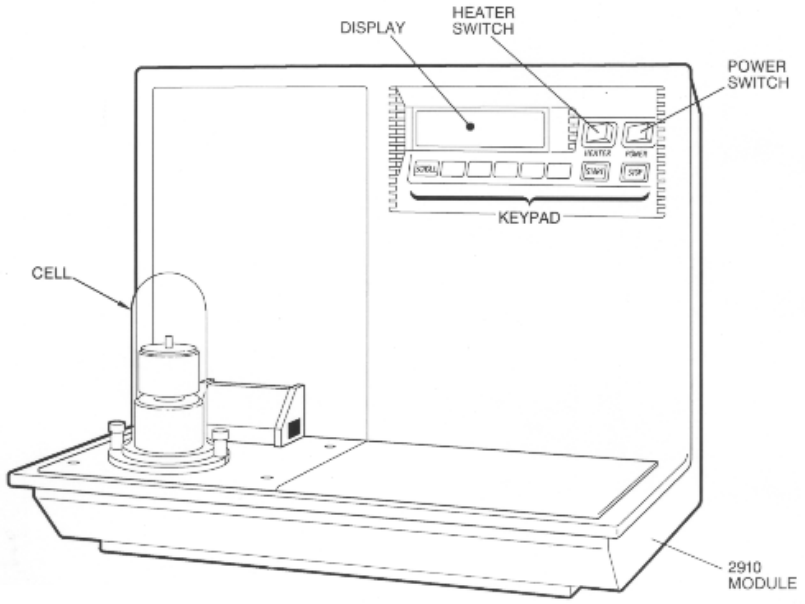


Figure 1.3
DSC 2910 with
Standard DSC Cell

Pressure DSC Cell

The Pressure DSC (PDSC) cell (Figure 1.4) is a DSC cell enclosed in a steel cylinder that can be pressurized to 7 MPa (1000 psig). In addition to performing the same measurements as the DSC cells, it can operate at elevated pressure or under vacuum. This ability to vary pressure as well as temperature provides the following:

- Resolution of overlapping peaks
- Determination of heats of vaporization and vapor pressure
- Reaction rates in controlled atmospheres
- Studies of pressure-sensitive reactions.

The Pressure DSC cell has two gas flow control valves, a three-way valve, a pressure gauge, a pressure release valve, and gas pressure fittings on the side. An 8.3 MPa (1200 psig) pressure relief valve is contained in the base of the cell.

NOTE:

The standard pressure cell (PN 900830.908) is designed for experiments using gases such as air, nitrogen, oxygen, helium, argon, carbon dioxide, and carbon monoxide. It is not designed for work in chlorine, bromine, or sulfur dioxide gas. A special version of the pressure cell (PN 900830.901) is available for work in hydrogen.

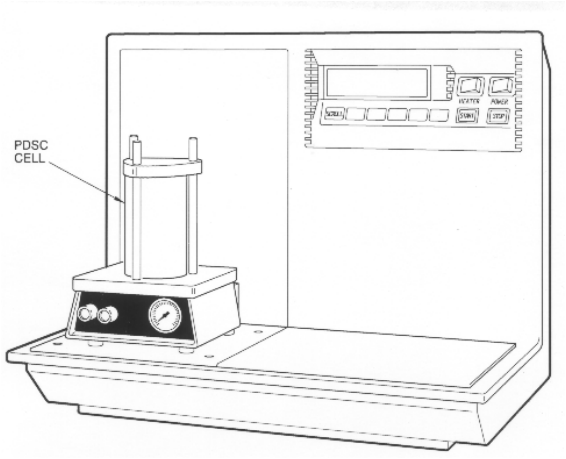


Figure 1.4
Pressure DSC Cell

1600°C DTA Cell

The 1600°C DTA Cell (Figure 1.5) is used to determine the temperatures of heat-related transitions at high temperatures. The sample and reference materials are placed in cups that sit on the tops of two thermocouple pedestals within the furnace tube of the 1600°C furnace. The thermocouples measure both the presence of transitions and the temperatures at which they occur.

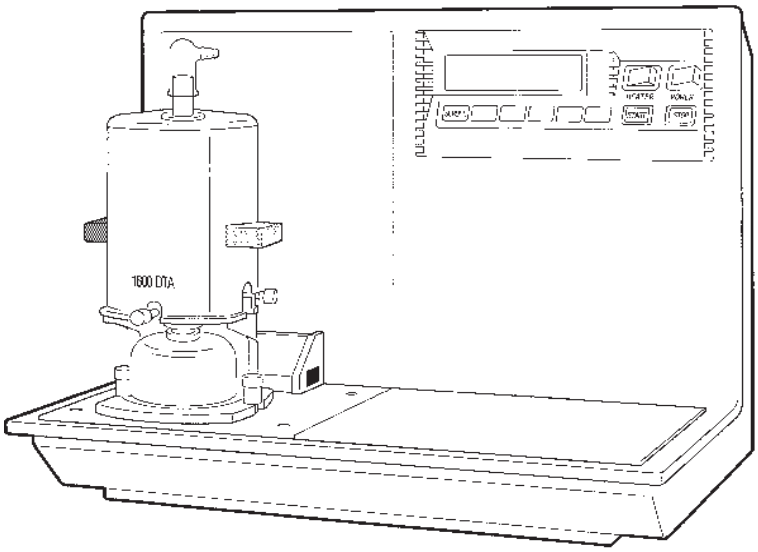


Figure 1.5
1600°C DTA Cell

Accessories

Sample Encapsulating Press

The TA Instruments Sample Encapsulating Press (Figure 1.6) is used to prepare encapsulated samples for DSC and PDSC experiments. It comes with two sets of dies, one for hermetic and one for non-hermetic sealing.

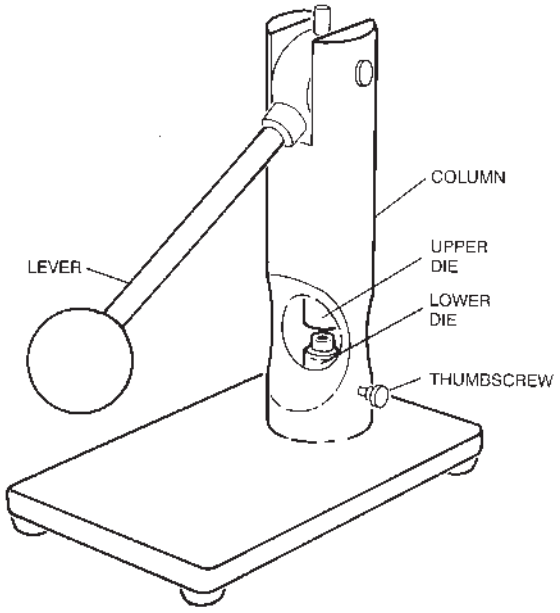


Figure 1.6 Sample Encapsulating Press

DSC Autosampler

The DSC Autosampler automatically loads sample and reference pans to the DSC, allows the programmed experiment to finish, then unloads the pans and begins the next experiment.

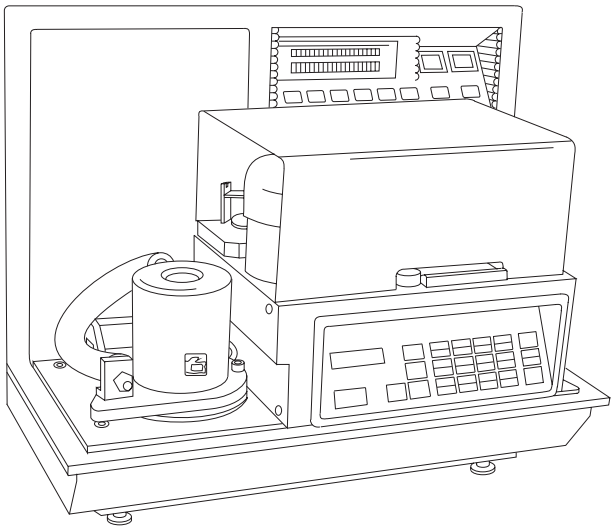


Figure 1.7
DSC Autosampler

Differential Photocalorimeter (DPC)

The DPC enables you to apply the principles of DSC to the measurement of chemical reactions initiated by high-intensity ultraviolet of visible light. Measurements include:

- Heat released by the sample and reference as they are exposed to radiation of known wavelength and intensity in a temperature-controlled environment.
- Physical properties, such as glass transition before and after exposure to radiation.

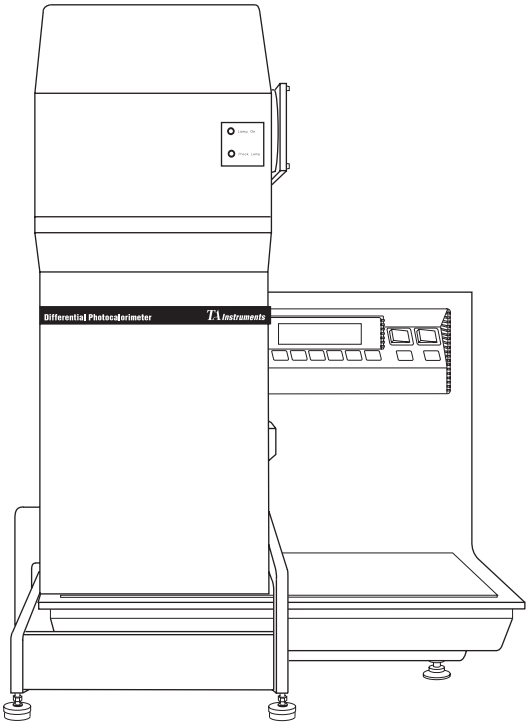


Figure 1.8 Differential Photocalorimeter

Accessories for Subambient Operation

The DSC 2910 can be operated at below-ambient temperatures using one of the cooling accessories such as the Liquid Nitrogen Cooling Accessory (LNCA), Refrigerated Cooling System (RCS), or the DSC Cooling Can.

Heat Exchanger

The heat exchanger works in conjunction with the LNCA to cool down samples on the 2910. The heat exchanger fits over the standard DSC cell.

LNCA

The LNCA (Figure 1.9) provides automatic and continuous programmed sample cooling within the range of -150°C to 725°C when used with the DSC heat exchanger installed on the DSC Cell. Heaters vaporize the liquid nitrogen in the LNCA tank. The cool gas is forced up and mixed with liquid nitrogen. The gas/liquid mix is delivered to the heat exchanger to cool the cell.

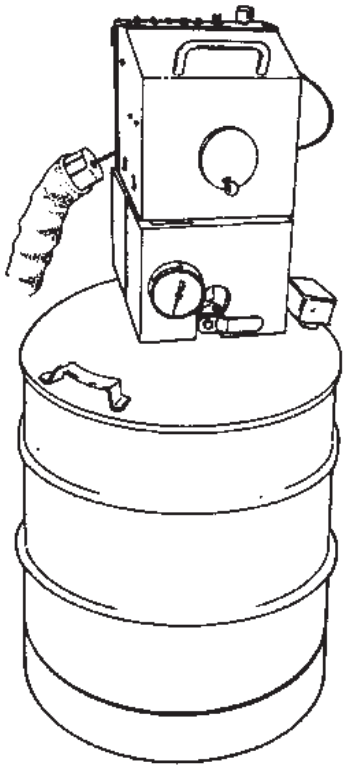


Figure 1.9
LNCA

Refrigerated Cooling System (RCS)

The Refrigerated Cooling System (RCS), which is used to cool DSC experiments, consists of a two-stage, cascade, vapor compression refrigeration system with an attached cooling head. The cooling head fits over the RCS-DSC cell for use with the DSC 2910. The RCS can be used for experiments requiring cooling within an operating range of -70°C to 400°C. The maximum rate of cooling depends on the temperature range of your experiment.

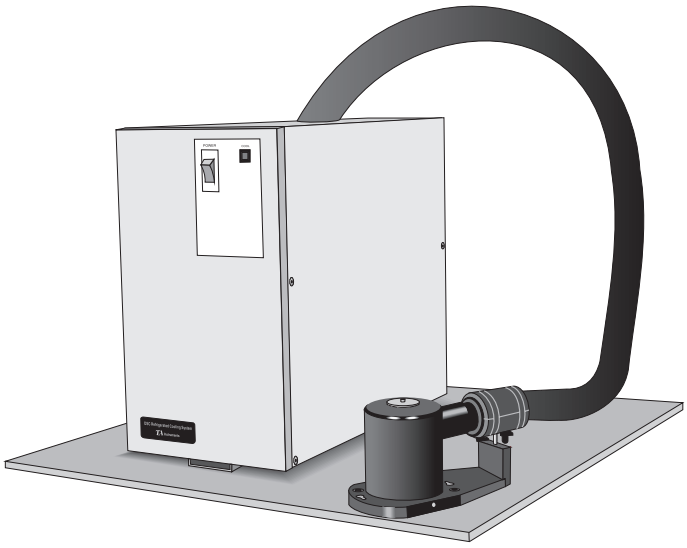


Figure 1.10
Refrigerated Cooling System

DSC Cooling Can

The DSC Cooling Can fits over the standard DSC cell and has a reservoir into which you can place coolant to cool the cells. Either quench cooling or manual programmed cooling can be performed. The manual programmed cooling requires operator maintenance of the coolant level in the reservoir.

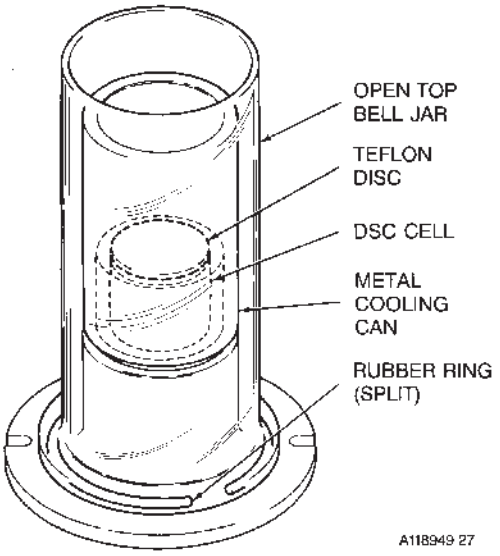


Figure 1.11
DSC Cooling Can

Specifications

Tables 1.2 through 1.6 contain the technical specifications for the DSC 2910 and its three cell types.

Table 1.2
DSC 2910
Specifications*

Dimensions	Depth 45.5 cm (18 in.) Width 58.5 cm (23 in.) Height 49.5 cm (19.5 in.)
Weight (approx.)	18 kg (40 lb)
Power	115 volts AC ±10% 50/60 Hz 1000 VA
Room Operating Temperature	15°C to 30°C

* Only values with tolerances or limits are guaranteed data. Values without tolerances are for information only.

Table 1.3
Standard DSC
Cell Specifications

Dimensions	Depth 13 cm (5.2 in.) Height 19 cm (7.3 in.)
Weight (approx.)	2.3 kg (5 lb)
Temperature range	Room temperature to 725°C (inert atmosphere above 600°C) as supplied; to -150°C with the LNCA and DSC Cooling Can; to -70°C with the RCS.
Cooling rate	Dependent on accessory used and temperature range
Sample size	0.5 to 100 mg (nominal)
Sample volume	10 mm^3 in hermetic pans
Sample pans	Various open or hermetically sealed
Atmosphere	Atmospheric to 266 Pa (2 torr); preheated dynamic gas purge (100 ml/min maximum)
Purge gases	Recommended: air, argon, helium, nitrogen, or oxygen
Typical flow rate	25-50 mL/min
Cell volume	2 cm^3

(table continued)

Table 1.3 continued

Temperature repeatability	±0.1°C
Differential thermocouples	CHROMEL®*-constantan (Type E)
Sample thermocouple	CHROMEL®-ALUMEL®* (Type K)
Control thermocouple (standard cells)	Platinel II**
Control thermocouple (hydrogen pressure cell)	CHROMEL®-ALUMEL®* (Type K)
Calorimetric sensitivity	3 µW (rms)
Constant calorimetric sensitivity	±2.5% from -100 to 500°C
Calorimetric precision	1% (based on metal samples)
Baseline noise	1.5 µW (rms)

* CHROMEL® and ALUMEL® are registered trademarks of Hoskins Manufacturing Company.

** Platinel is a registered trademark of Engelhard Industries.

Table 1.4
Pressure DSC Cell
Specifications

Dimensions	Length 21 cm (8.4 in.) Width 19 cm (7.4 in.) Height 24 cm (9.6 in.)
Weight (approx.)	9.1 kg (20 lbs)
Temperature Range	Room temperature to 725°C
Atmosphere	1.3 Pa to 7 MPa, constant pressure or constant volume
Dynamic gas purge	To 200 ml/min
Baseline noise	± 30 µW (rms)
Other specifications similar to those of the standard DSC Cell. Performance depends on pressure and atmosphere selected.	

Table 1.5
1600°C DTA Cell

Dimensions	Depth 14 cm (5.5 in.) Length 19 cm (7.3 in.)
Weight (approx.)	1.8 kg (4 lbs)
Temperature range	Ambient to 1600°C
ΔT sensitivity	0.001°C
Sample size	Up to 75 mm^3
Sample cups	Platinum micro (3 mm ID) Platinum macro (5mm ID) Alumina micro (3 mm ID)
Sample and reference thermocouples	Platinum-platinum/13% rhodium (Type R)
Control thermocouple	Platinum-platinum/13% rhodium (Type R)
Atmosphere	Static or controlled flow with inert or reactive gas or air
Pressure	Atmospheric to 266 Pa (2 torr)
Temperature precision	±2°C

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Unpacking/Repacking the 2910

NOTE: These instructions are also found as separate unpacking instructions in the shipping box.

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.

Unpacking the 2910

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

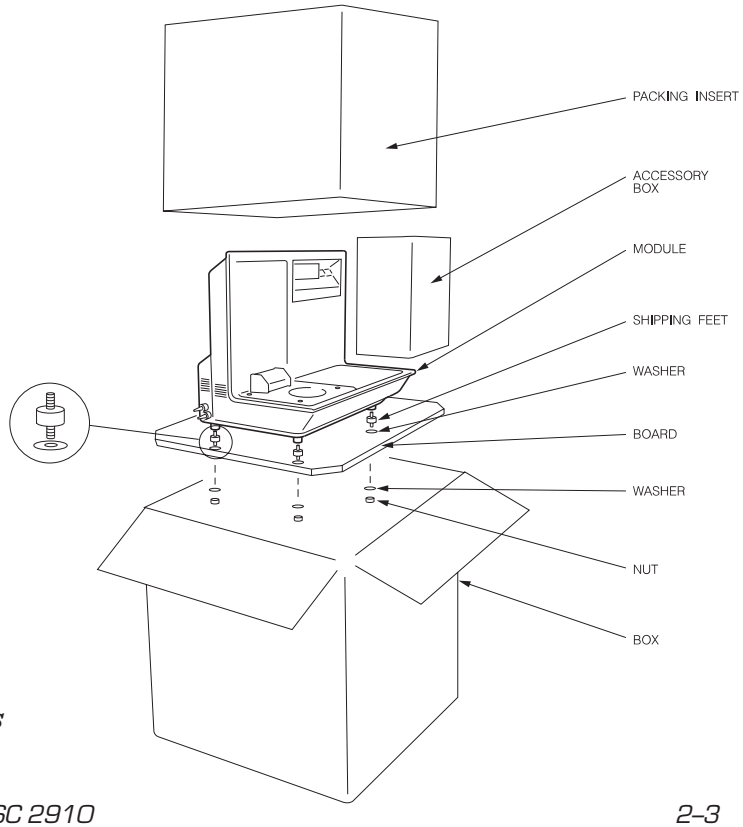


Figure 2.1
Shipping Boxes

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.**

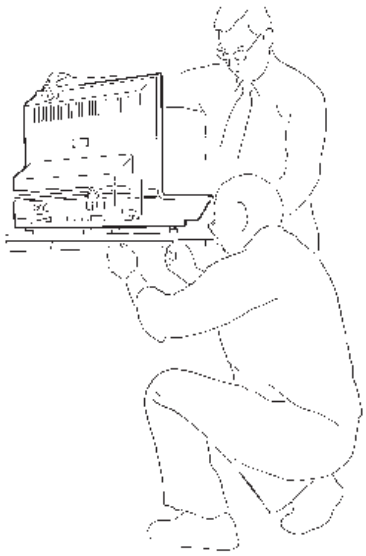


Figure 2.2
Removing the Plywood Board

5. While your assistant holds the unit, use a wrench to remove the two 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 two nuts and washers from the other side.

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

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

8. Have your assistant lift one side of the unit while you use a wrench to install two mounting feet (see Figure 2.3). Rotate the unit and install the two remaining mounting feet in the same manner.

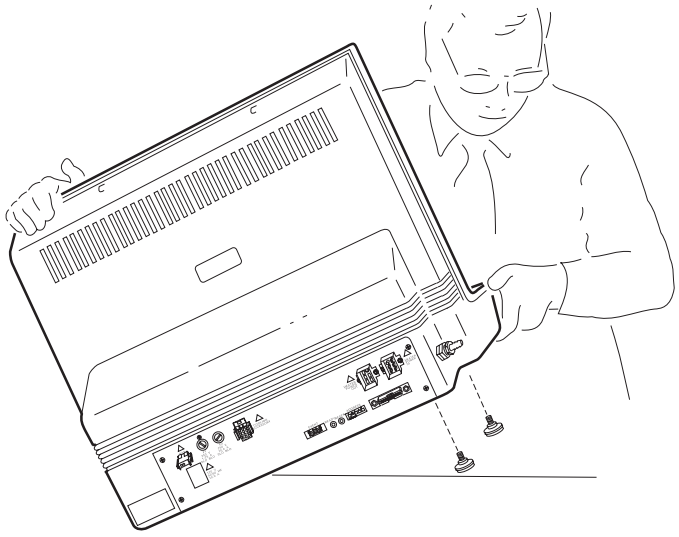


Figure 2.3 Installing the Mounting Feet

Repacking the 2910

To pack and ship your instrument, use the hardware retained during unpacking and reverse the instructions found on pages 2-3 to 2-5.

Installing the Instrument

Before shipment, the DSC 2910 is inpected 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:

- Inspecting the system for shipping damage and missing parts
- Connecting the instrument to a PC-based controller
- Connecting the gas and vacuum lines, accessories, and power cable
- Installing the desired cell type.

If you wish to have your DSC 2910 installed by a TA Instruments Service Representative, call for an installation appointment when you receive your instrument.

Inspecting the System

When you receive your DSC 2910, look over the instrument and shipping container carefully for signs of shipping damage, and 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.

The address for the TA Instruments office nearest you can be found in Appendix B of this manual.

Choosing a Location

Because of the sensitivity of DSC experiments, it is important to choose a location for the instrument using the following guidelines. The DSC 2910 should be:

In
- . . . a temperature-controlled area (15°C to 30°C is recommended).
- . . . a clean environment.
- . . . an area with ample working and ventilation space.

(Refer to the technical specifications in Chapter 1 for the instrument's dimensions.)

On
- . . . a stable work surface.

Near
- . . . a power outlet (115 Vac, 50 or 60 Hz, 15 amps). A step up/down line transformer may be required, if the unit is operated from a higher or lower line voltage.
- . . . your thermal analysis controller.
- . . . a compressed lab air and purge gas supply for use during cooling, subambient, and high temperature experiments.

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

NOTE: 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. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation. *...continued on next page*

NOTE: Run the following method to dry out the instrument:

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

Connecting Cables and Gas Lines

To connect the cables and gas lines, you will need access to the DSC 2910's rear panel. All directional descriptions 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 thumbscrews on all computer cables.

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

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

GPIB Cable

To connect the GPIB cable, follow these directions:

1. Locate the GPIB connector on the right rear of the DSC 2910 (see Figure 2.4).

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

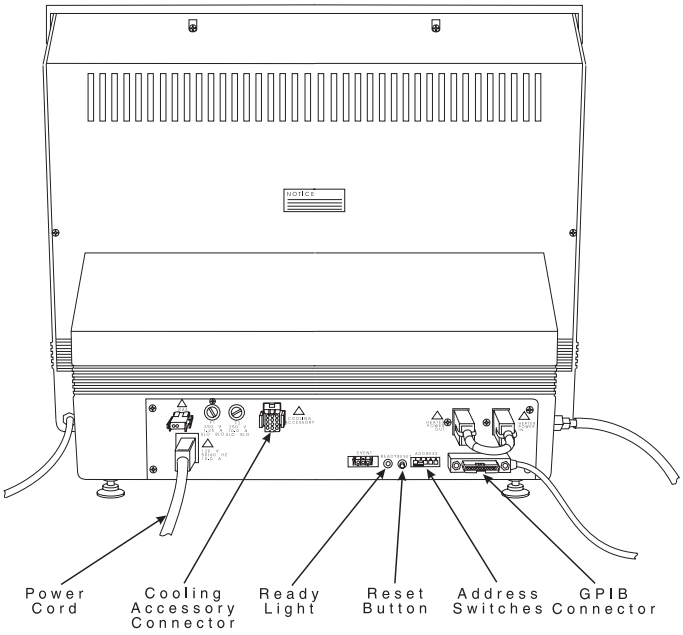


Figure 2.4
DSC 2910
Connector Panel

5. Select a unique address from 1 to 9 (one that is not used by any other instruments connected to your controller). Then use the binary address switches on the DSC 2910 connector panel to set the desired address (see Table 2.1). Figure 2.5 shows an instrument address of 7.

If you change the address after the instrument is powered on, you must press the Reset button on the instrument to enter the new address. Wait 30 seconds after releasing the Reset button, the green Ready light should begin to glow steadily. Then reconfigure the instrument with the controller to bring the instrument back online.

Table 2.1
Binary Address Settings*

Address	Switch Pattern 1 2 3 4 5
1	0 0 0 0 1
2	0 0 0 1 0
3	0 0 0 1 1
4	0 0 1 0 0
5	0 0 1 0 1
6	0 0 1 1 0
7	0 0 1 1 1
8	0 1 0 0 0
9	0 1 0 0 1
*0 = OFF; 1 = ON	

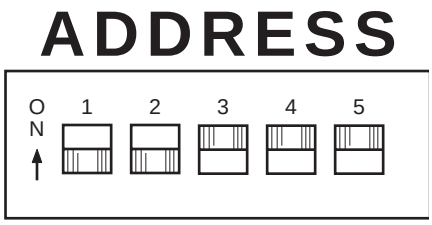


Figure 2.5
Binary Address Switches (Showing an Address of 7)

Purge, Vacuum, and Cooling Gas Lines

NOTE:

The PURGE, VACUUM, and COOLING GAS fittings on the DSC 2910 do not connect to the PDSC Cell. The PDSC Cell has its own direct gas line fittings. Instructions for connecting gas lines to the PDSC Cell are given in the section on the Pressure DSC Cell. The instructions given here are relevant to DSC and 1600°C DTA cell types.

PURGE Line

The PURGE typically is used to control the environment around the sample.

1. Locate the PURGE fitting on the right side of the DSC 2910 back (see Figure 2.6).

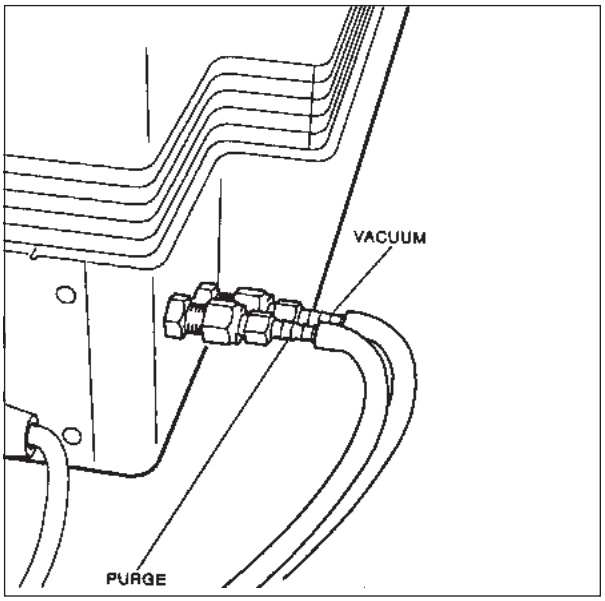


Figure 2.6 PURGE and VACUUM Fittings

2. Make sure your purge source is regulated between 5 and 30 psi and connected to a flowmeter to regulate flow up to 200 ml/min.

WARNING

Use of any explosive gas as a purge gas is dangerous and is not recommended for the DSC 2910.

CAUTION:

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

3. Connect a 6.2 mm (1/4-inch) I.D. flexible tubing purge line to the PURGE fitting.

VACUUM Line

The VACUUM line is used to help minimize the build up of moisture in the cell during cooling experiments and to remove gases evolved from samples during experiments.

1. Locate the VACUUM fitting on the right side of the DSC 2910 back (see Figure 2.3).

2. Connect a 6.2 mm (1/4-inch) I.D. flexible tubing vacuum line to the VACUUM fitting.

NOTE:

To minimize moisture build-up during subambient experiments, supply a dry nitrogen purge to the vacuum line using a rate of 100 – 150 ml/min.

COOLING GAS Line

To prevent vibration of the bell jar, use a split O-ring when you use cooling gas with the standard DSC Cell. A split O-ring is provided with the DSC Cooling Can. If you do not have a split O-ring, you can order one from TA Instruments or modify the one that comes with the DSC Cell. By cutting a small (1 cm) section out of the DSC cell O-ring before placing it under the bell jar, you can prevent possible vibration when using the cooling gas with the DSC.

NOTE: The COOLING GAS line is not operational when the instrument is set to a DTA mode. This is to prevent the cap on the furnace tube from popping off when the COOLING GAS valve is opened.

Connect the COOLING GAS line as follows:

1. Locate the COOLING GAS fitting, a 6.2 mm (1/4-inch) compression fitting on the left side of the DSC 2910 back, marked with a 120 psi maximum warning label (see Figure 2.7).

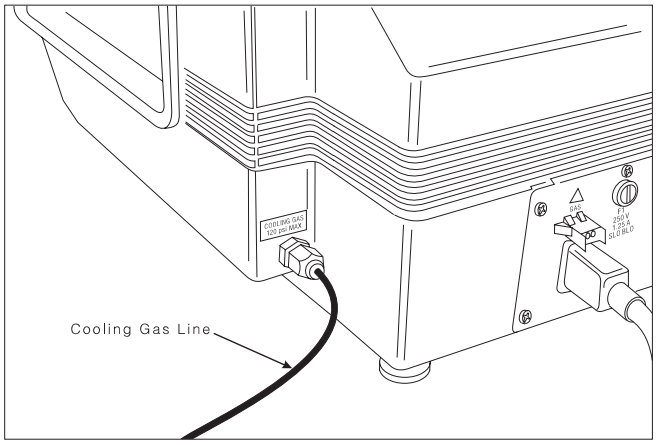


Figure 2.7 COOLING GAS Fitting

2. Make sure your cooling gas source is regulated between 20 and 120 psi.

♦ ***CAUTION:***

The COOLING GAS line feeds into a pressure-regulated valve that is set to 15 psi. The source pressure setting should not go below this value.

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

Power Cable

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

1. Make sure the DSC 2910 POWER switch (see Figure 2.8) is in the OFF (0) position.

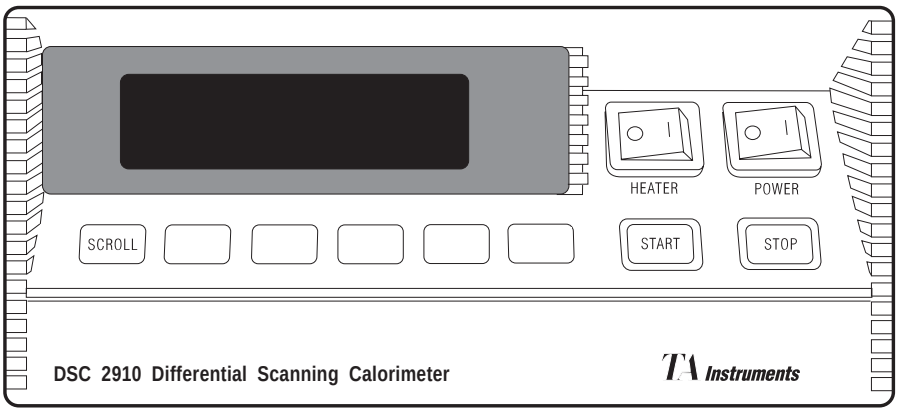


Figure 2.8
DSC 2910
POWER Switch

2. Plug the power cable into the DSC 2910.

♦ ***CAUTION:*** **Before plugging the DSC 2910 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 or step down/up transformer.

Installing the Standard DSC Cell

To install the standard DSC cell on the DSC 2910, follow the instructions found in this section. When unpacking the cell from its original container, remove and discard all packing material, such as tape and polyethylene film.

1. Remove the bell jar from the cell you are about to install (see Figure 2.9).

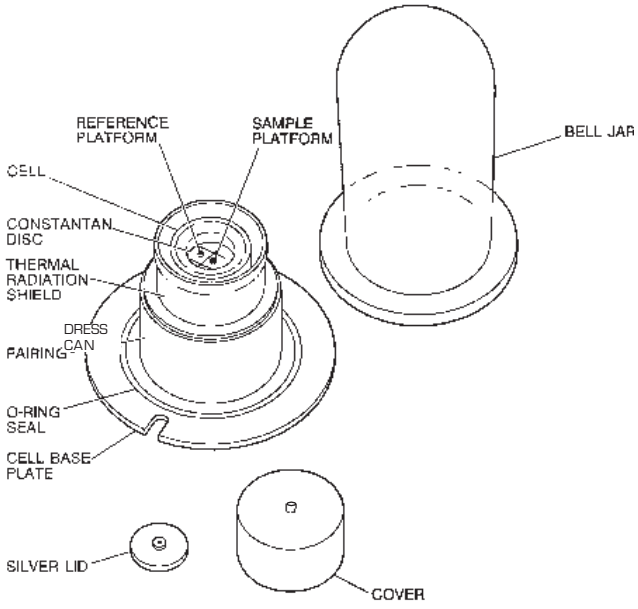


Figure 2.9
Standard DSC Cell

2. Place the DSC Cell over the alignment pins on the 2910 instrument (Figure 2.10) so that the green dot on the cell base plate faces to the front. Press down on the cell base plate to seat the cell connector.

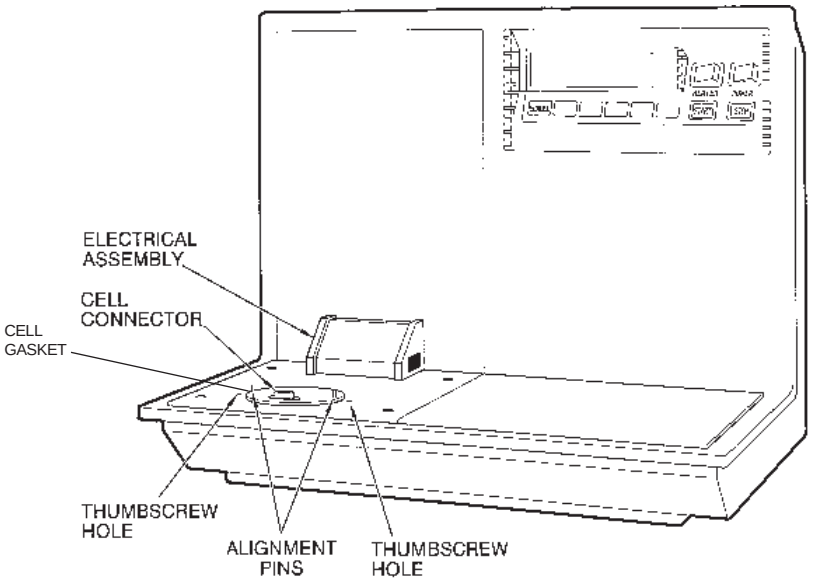


Figure 2.10
DSC 2910
Cell Base Connectors

3. Install the two hold-down thumbscrews shipped in the 2910 instrument accessory kit. Finger-tighten them slowly and evenly to ensure proper gas line connections.

4. Place the silver lid, cell cover, and bell jar over the cell. The knob on the silver lid should be pointing up.

Installing the Pressure DSC Cell

To install the Pressure DSC (PDSC) Cell on the DSC 2910, follow the instructions below, and refer to Figures 2.10 and 2.11.

♦ ***CAUTION:***

Do not remove the white, fibrous insulation from inside the cell cover. Refer to the MSDS sheet supplied with the Pressure cell for necessary precautions.

!WARNING

Turn the HEATER switch to the off (O) position when installing or removing cells to reduce the possibility of an electrical shock.

1. If you are removing the cell from its original container:

   a. Turn the three thumbscrew bolts clockwise until they drop into the PDSC cell base.

   b. Remove the top plate and discard the packing material from inside the cell, but *do not* remove the white insulation from inside the cell cover.

   c. Install the right angle fitting on the pressure release valve on the back of the PDSC. Tighten the right angle fitting until it points toward the inlet fitting.

2. Screw the two PDSC baseplate alignment pins (supplied with the PDSC cell) into the thumbscrew holes in the 2910 instrument (see Figure 2.10).

♦ ***CAUTION:*** **The baseplate alignment pins are used to protect the cell connector from damage when you install the PDSC cell onto the 2910 instument.**

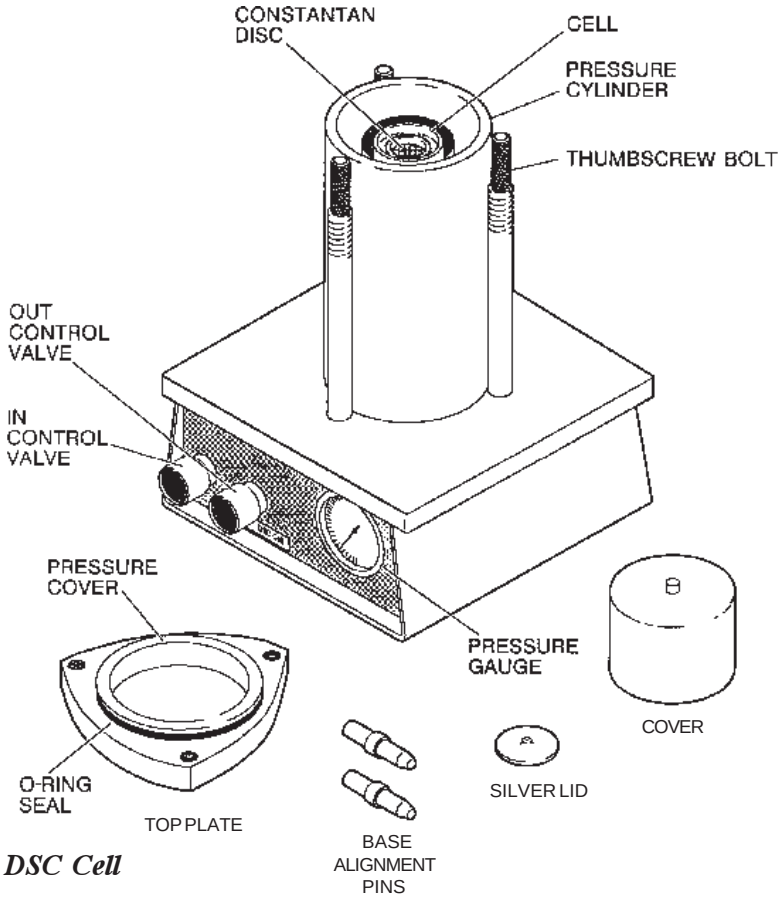


Figure 2.11
The Pressure DSC Cell

3. Connect a sufficient length of 0.32-mm (0.125-inch) tubing from a pressure regulator on your pressurized gas source (nitrogen, air, oxygen, etc.), pressure-regulated up to 7 MPa (1000 psig) to the appropriate connector on the back of the PDSC cell:

   • If you wish to purge gas through the cell during your experiment, connect the supply gas line to the OUT fitting on the

back of the cell and connect a flowmeter to the IN fitting. The reason for this reversal of flow is to preheat the purge gas before it enters the cell chamber.

- If you do not plan to use a purge, connect the supply gas line to the IN fitting on the back of the cell and close the OUT valve to allow pressure puild-up; no connections to the OUT fittings are necessary unless you wish to run an exhaust line out of your lab.

♦ ***CAUTION:*** **If oxygen is used, be certain to use fittings, gauges, and tubing that are oxygen-rated.**

The regulator you choose should have two gauges: one to monitor source pressure and one to monitor the regulator output pressure. The regulator should be rated to withstand the source pressure; its output should cover the experimental range up to 7 MPa (1000 psig).

!WARNING **DO NOT connect the PDSC directly to a pressurized gas source without using an appropriate regulator.**

!WARNING **The tubing must be of sufficient strength to withstand the pressure to be used in your experiments.**

!WARNING **Hydrogen can be used only in a special version of the pressure cell (PN 900830.901). Hydrogen gas should be used with extreme care. It is highly flammable when exposed to flame or oxidizing materials. When using hydrogen in the pressure DSC cell, the cell should be initially purged thoroughly with helium before introducing hydrogen. At the end of the experiment, or if purging the cell during the experiment, the hydrogen in the cell should be vented to an exhaust hood and repurged with helium prior to opening the pressure container.**

Installing the 1600°C DTA Cell

Before you install your 1600°C DTA Cell (Figure 2.16), check the accessory kit that came with the cell to ensure that it contains the following items:

- 1 adapter, 90-degree bend standard
- 1 spatula, style B
- 1 package of platinum liners
- 1 package of alumina liners
- 1 package of macro cups
- 1 aluminum oxide sample (with Material Safety Data Sheet)
- 1 9/64-inch hex wrench (modified)
- 1 furnace alignment tool
- 1 furnace tube
- 1 3/32-inch hex wrench (modified)
- 2 thumbscrews
- 1 silver crystal sample (with Material Safety Data Sheet).

Installation of the 1600°C DTA Cell consists of these steps, which are detailed in the pages that follow:

!WARNING

Turn the HEATER switch to the off (O) position when installing or removing cells to reduce the possibility of an electrical shock.

(1) Installing the furnace tube
(2) Installing the thermocouple assembly
(2) Installing the cell on the DSC 2910
(3) Aligning the cell furnace.

You should become familiar with the cell furnace alignment procedure before you attempt to run experiments with the DTA Cell.

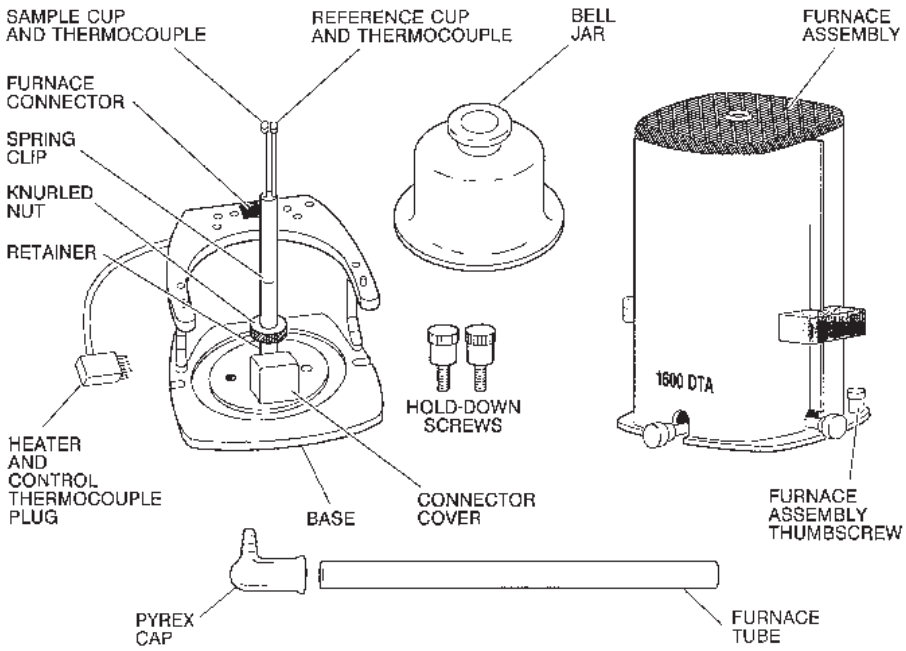


Figure 2.12
Parts of the
1600°C DTA Cell

Installing the Furnace Tube

After you have unpacked all the parts of your DTA cell. The first step in putting it together is to install the furnace tube. Place the DTA cell on a stable surface and perform these steps:

1. Remove the furnace from the cell by unscrewing the two furnace assembly thumbscrews (see Figure 2.13).

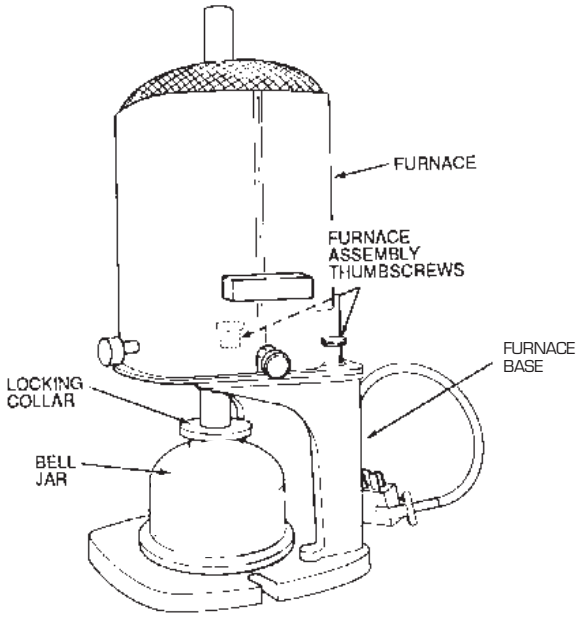


Figure 2.13
1600°C DTA Cell

2. Remove the DTA bell jar from the cell.

3. Slide the straight end (not the tapered end) of the furnace tube carefully into the opening on the top of the bell jar and firmly seat it.

4. Tighten the bell jar locking nut using an Allen wrench. Set the bell jar and furnace tube assembly to one side while you proceed with the installation of the thermocouple and cell.

Installing the Thermocouple Assembly

NOTE:

The 1600°C DTA Cell thermocouple assembly (PN 900410.901) is the cell's temperature sensor. It consists of a matched pair of platinum-platinum/13% rhodium thermocouples mounted in a ceramic post. Because of its fragility, the thermocouple assembly is packaged separately from the cell and must be installed by the operator.

1. Remove the thermocouple assembly (see Figure 2.14) from its plastic shipping container and check it for damage. If the assembly is damaged, notify TA Instruments immediately.

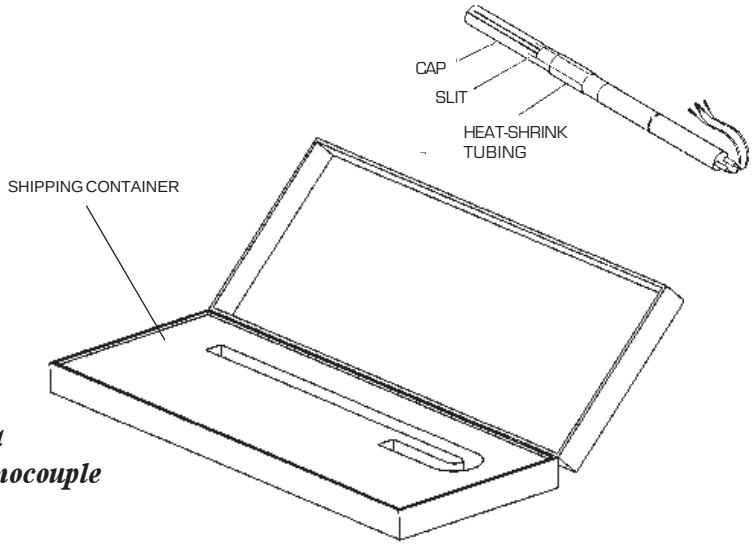


Figure 2.14 DTA Thermocouple Assembly

2. Remove the caution label from around the thermocouple assembly.

3. Using a knife or razor blade, carefully cut and remove the heat-shrink tubing from the thermocouple assembly.

4. Carefully lift the slitted bottom edges of the plastic cap to loosen it from the ceramic post, and slide the cap off the post.

5. Insert the thermocouple wires through the hole in the knurled nut.

6. Carefully insert the thermocouple assembly (with the knurled nut), wire-lead end first, into the thermocouple support, slipping the wire leads through the slot in the support (see Figure 2.15).

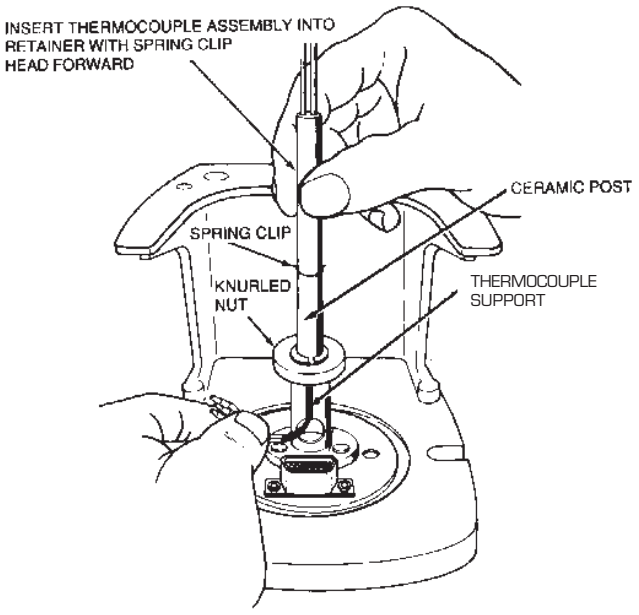


Figure 2.15 Installing the Thermocouple

7. Rotate the assembly so that the head of the spring clip on the ceramic post faces toward you.

8. Gently push the thermocouple down until it will go no further. Lock the post in position by tightening the knurled nut.

NOTE: The thermocouple distance is set at TA Instruments before shipment. Use Figure 2.16 as a guide if you should need to reset the thermocouple distance.

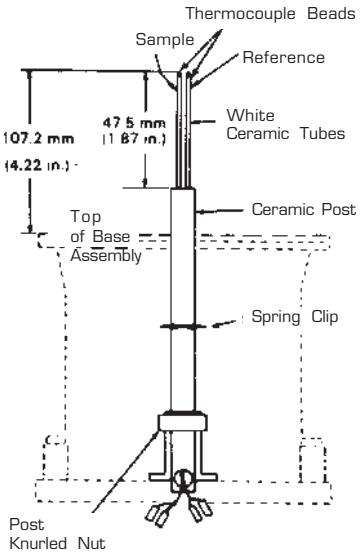


Figure 2.16 Guidelines to Adjust the Thermocouple Distance

9. Reset the thermocouple distance, if necessary, as follows:

   a. Measure the distance from the thermocouple beads (at the tops of the white ceramic tubes) to the top of the ceramic post. The distance should be 47.5 mm (1.87 inches).

   b. If the distance is incorrect, remove the spring clip from the ceramic post, carefully position the thermocouples at the correct distance, and replace the spring clip.

10. Loosen the knurled nut securing the ceramic post, and position the ceramic post so that the distance from the thermocouple tips to the top of the DTA furnace base is 107.2 mm (4.22 inches). Then tighten the knurled nut.

11. Pull off the connector cover (see Figure 2.13) and remove, but *do not discard* the insulation from around the connectors. Using long-nosed pliers, plug the thermocouple wires into the connector pins as shown in Figure 2.17. Repack all of the insulation around the connector pins, and replace the connector cover.

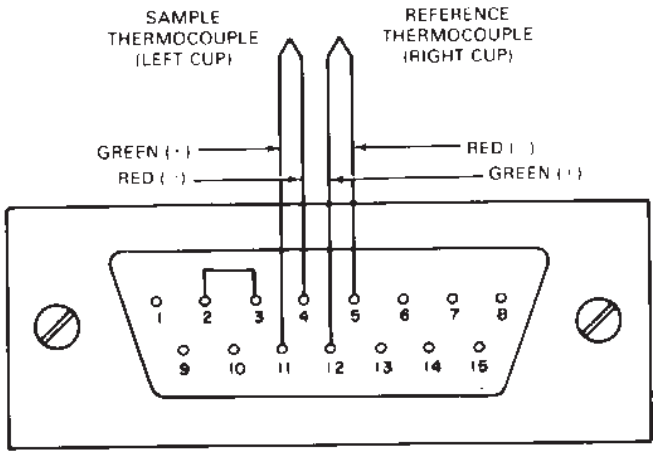


Figure 2.17
DTA
Thermocouple
Connections

12. Gently lower the furnace tube and bell jar assembly over the thermocouple.

♦ ***CAUTION:***

Be very careful not to damage the thermocouple as you lower the furnace tube and bell jar. If the thermocouple becomes damaged, you will need to replace it.

Now you are ready to install the DTA cell onto the instrument.

Installing the Cell

Refer to Figures 2.12, 2.13, and 2.18, while installing the 1600°C DTA Cell with the furnace tube in place.

1. Place the DTA furnace base on the 2910 instrument, using the alignment pins on the instrument as guides. Firmly seat the furnace base on the instrument.

2. Secure the furnace base to the 2910 instrument (and the bell jar to the furnace base) by firmly and evenly tightening the two larger hold-down screws supplied with the DTA.

♦ ***CAUTION:*** **Do not use the hold-down screws that come with the DSC cell.**

3. Slowly lower the furnace over the furnace tube, and using the guide pins on the furnace base, seat the furnace on the furnace base (see Figure 2.18).

♦ ***CAUTION:*** **If the furnace assembly does not slide easily over the furnace tube, call your TA Instruments service representative. Do not force the furnace assembly over the furnace tube.**

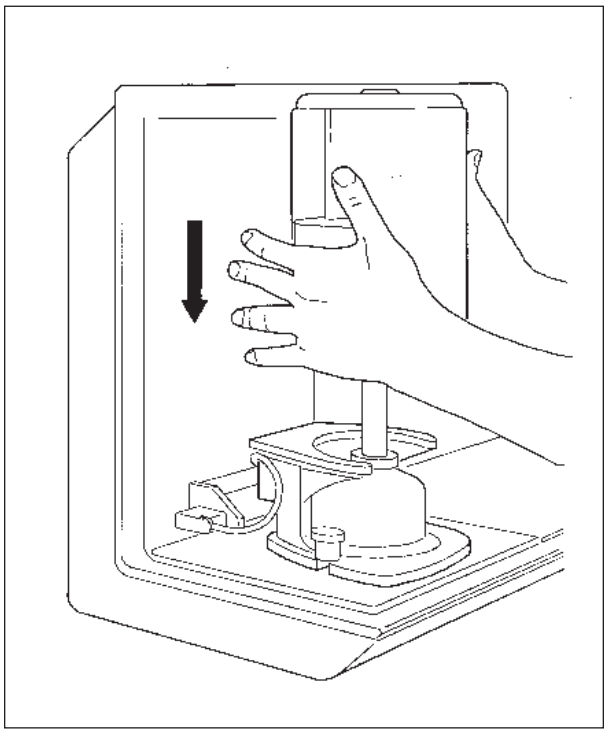


Figure 2.18 Installing the DTA Furnace Assembly

4. Firmly and evenly tighten the two furnace assembly thumbscrews. Ensure that the assembly is tightly secured and does not wobble on the furnace base.

5. Plug the DTA cell power cable into the left receptacle on the front of the instrument (see Figure 2.19). The DTA cell is grounded through this cable.

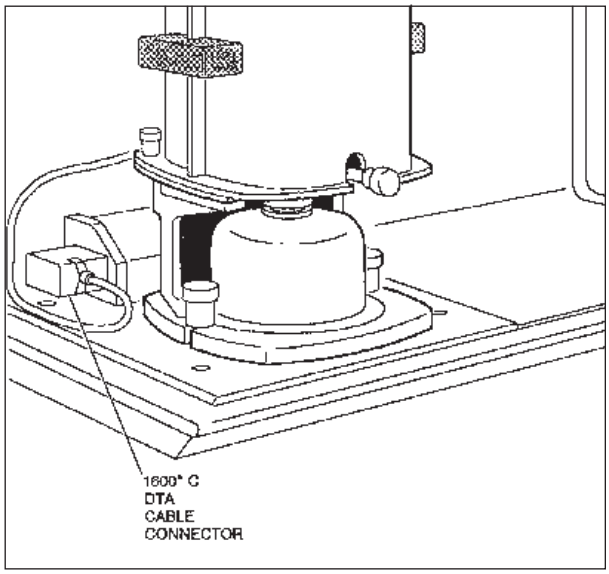


Figure 2.19 Attaching the DTA Cell to the Instrument Base

NOTE: The DTA transformer is not required for use with the DSC 2910.

Aligning the DTA Cell Furnace

After you install the 1600°C DTA cell for the first time, you must align the cell furnace around the furnace tube to ensure proper heating and cell performance during experiments. If you later remove and reinstall the DTA cell, the alignment should not be affected, but you may wish to check it as described in step 2 below.

Three adjustments to the furnace core are necessary:

- Right-to-left adjustment
- Front-to-rear adjustment
- Angular adjustment (adjusting the angle of the furnace core so that it is parallel to the furnace tube).

You will need to use the calibration rod in the DTA Installation Kit to perform the alignment. A flashlight or other source of light is also helpful in determining the clearance between the cell furnace and furnace tube.

1. Center the furnace tube around the thermocouple assembly, and tighten the locking collar firmly using the 2.4 mm (3/32-inch) hex wrench shipped in the DTA accessory kit.

2. Look down through the top of the cell and determine if the furnace tube is roughly in the center of the furnace. There must be equal clearance between the cell furnace and the furnace tube.

3. If the furnace tube is not centered within the furnace, turn the *x* and *y* screws (see Figure 2.20) in the appropriate direction (according to Table 2.2) to center the furnace around the furnace tube.

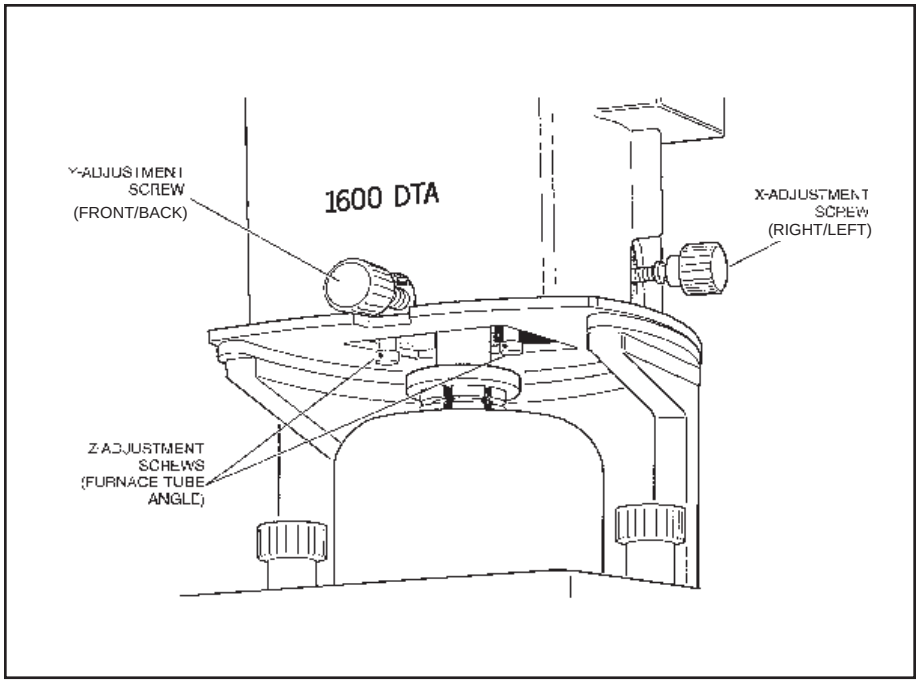


Figure 2.20
Location of the Adjustment Screws

Table 2.2
Using the X- and Y-Adjustment Screws

Adjustment Screw	Direction Turned*	Effect
x-adjustment screw	CW	Moves the furnace core to the left.
	CCW	Moves the furnace core to the right.
y-adjustment screw	CW	Moves the furnace core away from you.
	CCW	Moves the furnace core toward you.
*CW = clockwise; CCW = counterclockwise		

NOTE: Earlier versions of this cell, identified by the presence of a lockdown nut, will work opposite of the above chart. CW then becomes CCW and CCW would become CW.

4. Place the calibration rod in the space between the furnace core and the furnace tube. You should be able to move the calibration rod up and down freely in this space.

NOTE: Always move the calibration rod perpendicular to the furnace base when you check the furnace core and tube clearance. Do not pull the rod around the space.

If the rod does not move freely, the furnace core is not parallel to the furnace tube; there is not equal clearance between the furnace core and the furnace tube along the entire length of the calibration rod. You will need to make adjustments to the angle of the furnace core, as explained in step 5.

5. Use the two *z*-adjustment screws (see Figure 2.22) to correct the angle, or lean, of the furnace core. Refer to Tables 2.3 and 2.4 to determine which screw to adjust and the direction it should be turned.

Table 2.3 Using the Z-Adjustment Screw

Adjustment Screw	Direction Turned*	Effect
Left *z* screw	CCW	Moves the left side of the furnace core up
	CW	Moves the left side of the furnace core down
Right *z* screw	CCW	Moves the right side of the furnace core up
	CW	Moves the right side of the furnace core down

*CW = clockwise CCW = counterclockwise

Table 2.4 Z Screw Adjustment Guidelines

Rod sticks at:	Move left *z* screw:	Move right *z* screw:
12 o'clock	CW	CW
3 o'clock	CW	CCW
6 o'clock	CCW	CCW
9 o'clock	CCW	CW

*CW = clockwise CCW = counterclockwise

Adjusting the *z* screws may affect the *x* and *y* adjustments already made. Whether you need to readjust the *x* and *y* screws depends on how much the *z* screws were adjusted.

6. Continue with the following two-step sequence until the calibration rod enters and leaves the space freely without touching:

   a. If the rod touches, adjust the *z* screws as necessary (see Tables 2.3 and 2.4).

   b. Adjust the *x* and *y* screws if necessary. (Check using the short end of the calibration rod, or determine visually.)

Installations for Subambient Operation

The 2910 standard DSC cell can be operated at subambient conditions using any one of the following cooling accessories:

- Liquid Nitrogen Cooling Accessory (LNCA) with the DSC Heat Exchanger
- DSC Cooling Can
- Refrigerated Cooling System (RCS).

This section describes how to install the Cooling Can on the DSC 2910. The installation of the LNCA and the RCS with the DSC 2910 can be found in the literature accompanying those accessories.

Installing the DSC Cooling Can

The DSC Cooling Can is a metal can that fits over the standard DSC cell. Coolant is placed in a reservoir in the top of the can. An open-top bell jar, an insulation disc, and a split O-ring are included with the Cooling Can.

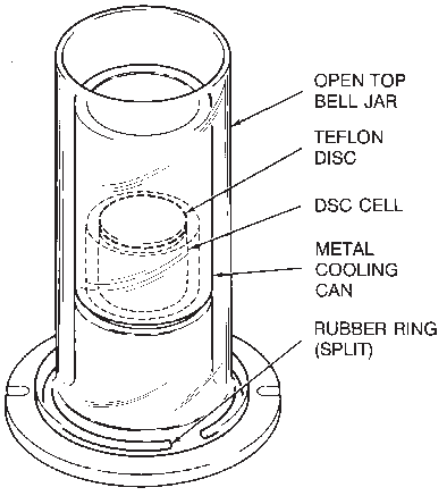


Figure 2.21
DSC Cooling Can

The installations for quench and programmed cooling are the same, with one exception: the insulation disc is used for programmed cooling *only*. The DSC Cooling Can can be used for programmed cooling once the insulation disc is installed. Be sure to determine which type of cooling you plan to use before you install this accessory. If you prepare the insulation disc according to the instructions on the next page, it may be removed at a later date to allow for quench cooling.

The components installed in the following steps are in the parts bag shipped with the DSC Cooling Can.

1. Remove the bell jar from the DSC Cell. Remove the original O-ring and replace it with the split O-ring shipped with the DSC Cooling Can.

2. If you plan to do *programmed* cooling experiments with the DSC Cooling Can, first punch a 2-cm hole in the center of the insulation disc. This allows you to remove the disc from the can later by prying up the edge of the hole with a tool. Place the insulation disc inside the can by turning the cooling can upside down, putting the disc into the can, and pushing the disc until it snaps into place (see Figure 2.22).

♦ ***CAUTION:***

Once the Teflon disc is installed, you cannot remove it; the DSC Cooling Can will be set up permanently for programmed cooling.

Teflons softens at 325°C.

If you plan to do *quench* cooling experiments with the DSC Cooling Can, *do not* install the insulation disc.

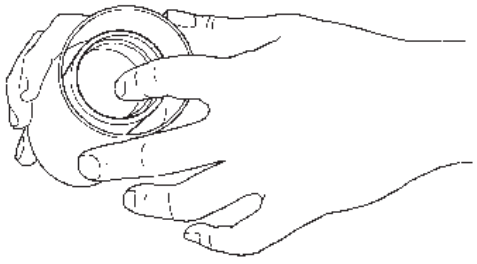


Figure 2.22 Installing the Insulation Disc in the DSC Cooling Can

3. Place the DSC Cooling Can over the DSC Cell.

4. Place the open-top bell jar over the DSC Cooling Can.

NOTE: When running subambient experiments, use a dry nitrogen purge through the vacuum port (100 – 150 ml/min) to eliminate moisture buildup inside the cell.

Starting the 2910

1. Check all connections between the DSC 2910 and the controller. Make sure each component is plugged into the correct connector.

2. Press the instrument POWER and HEATER switch to the ON position. The first screen to appear will display the results of the internal confidence test, which is run each time you power on the unit.

NOTE: The HEATER and POWER indicator lamps may flicker under low AC voltage conditions.

3. Watch the instrument display during the confidence test for any error messages that may be indicated. If an error occurs, make a note of the test number in which the error occurred and call TA Instruments for service.

After the confidence test, 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, then the standby display, shown in Figure 2.23.

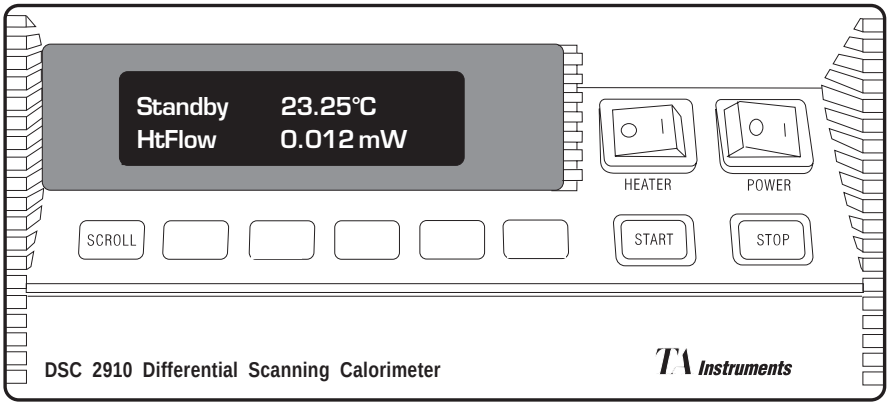


Figure 2.23
DSC 2910
Standby Display

NOTE: Instruments should warm up for at least 30 minutes before performing expeirments.

4. Bring the instrument online with the TA controller.

Shutting Down the 2910

Before you decide to power down your instrument, consider the following:

- All of the components of your thermal analysis system are designed to be left on for long periods.
- The electronics of the instrument and the controller perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, 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 you do need to power down your DSC 2910 for any reason, simply press the POWER and HEATER switches to the OFF position.

CHAPTER 3: Running Experiments

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Overview

This chapter gives instructions on how to run experiments with the DSC 2910 and each of the cell types:

- Differential Scanning Calorimeter (DSC)
- Pressure Differential Scanning Calorimeter (PDSC)
- 1600°C Differential Thermal Analyzer (DTA).

To obtain accurate results, follow the procedures carefully, and check the calibration periodically (*e.g.*, once a week).

Only the instructions necessary for running experiments are given in this chapter; explanations of terminology and how the instrument operates are given in Chapter 4, "Technical Reference."

Before You Begin

Before you set up an experiment, ensure that the desired cell, the DSC 2910, and the TA controller have been installed properly. Make sure you have:

- Made all necessary cable connections from the DSC 2910 to the TA controller
- Connected all gas lines
- Installed the desired cell onto the DSC 2910 (see Chapter 2)
- Powered up each unit
- Installed all appropriate options
- Configured the instrument online with the controller

- Become familiar with controller operations
- Calibrated the cell, if necessary (refer to the section below on calibration).

Calibrating the DSC

To obtain accurate experimental results you should calibrate each standard DSC and pressure DSC cell when you first install it. Once the initial calibrations are done, you can save the resulting data files and reuse them when needed. For the best results, however, you should recalibrate periodically.

Perform calibration runs that encompass the temperature range you plan to use in your experiments. If you change the general temperature range of your experiments later, you may wish to recalibrate within the new range.

For precise experimental results you will need to generate a new calibration file whenever you change one of the following parameters:

- Ramp rate (selected in the thermal method)
- Purge gas
- Cooling technique (LNCA, RCS, or DSC Cooling Can)
- Pressure (in PDSC experiments)
- First use of the cell.

However, an acceptable alternative is to use a previous calibration, if the conditions are sufficiently similar to those of the experiments you plan to run.

NOTE: Calibration is performed in the instrument's calibration mode, which is accessed through the controller.

Calibration consists of several different types of procedures specific to each cell, which are described briefly below. For more details on performing each type of calibration refer to the instructions in the *Thermal Solutions/Advantage User Reference Manual*.

Baseline Slope and Offset Calibration

The baseline slope and offset calibration needs to be performed separately for each cell. This calibration involves heating an empty cell through the entire temperature range expected in subsequent experiments. The results may look similar to Figure 3.1 on the next page. This figure shows two example heat flow curves for an empty standard DSC cell run from 25 to 400°C. Ideally, the heat flow signal should be zero, since there is no sample in the cell, and it should have minimum slope. The calibration program is used to calculate the slope and offset values needed to flatten the baseline and zero the heat flow signal.

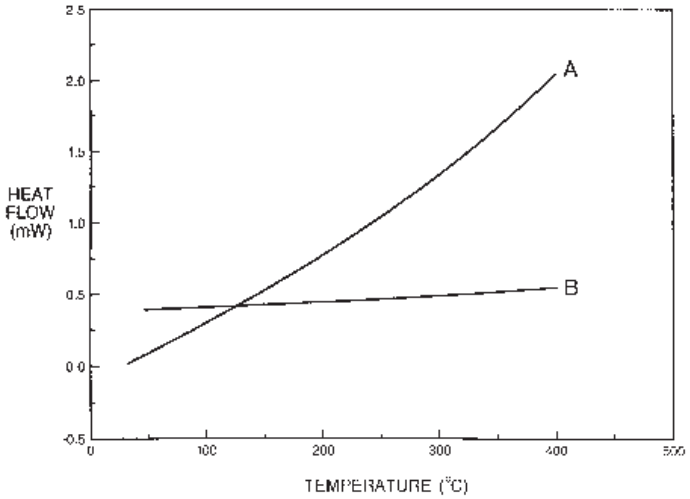


Figure 3.1
Baseline Slope Calibration

Cell Constant Calibration

This calibration is based on a run in which a standard metal (*e.g.*, indium) is heated through its melting point. The calculated heat of fusion is compared to the theoretical value. The cell constant is the ratio between these two values. The onset slope, or thermal resistance, is a measure of the temperature drop that occurs in a melting sample in relation to the thermocouple. Theoretically, a standard sample should melt at a constant temperature. As it melts and draws more heat, a temperature difference develops between the sample and the sample thermocouple. The thermal resistance between these two points is calculated as the onset slope of the heat flow versus temperature curve on the front of the melting peak. The onset value is used for kinetic and purity calculations to correct for this thermal resistance.

Temperature Calibration

Temperature calibration is based on a run in which a temperature standard (*e.g.*, indium) is heated through its melting point. The recorded melting point of this standard is compared to the known melting point, and the difference is calculated for temperature calibration. The same file used for the cell constant calibration can be used for this calibration.

In addition, you can use up to four other standards to calibrate temperature. If you use one pair of known and observed points, the entire curve is offset, or shifted, to the actual melting point. If you use multiple standards, the temperature is corrected by a cubic spline fit. The multiple-point temperature calibration is more accurate than the one-point calibration.

Running a DSC Experiment

Experimental Procedure

All of your DSC experiments will have the following general outline. In some cases, not all of these steps will be performed.

- Selecting and preparing a sample. This involves preparing a sample of the appropriate size and weight, selecting the pan type and material, and encapsulating the sample in the pan.
- Loading the sample pan (and a similarly prepared empty reference pan) into the cell.
- Entering experiment information through the TA controller (sample and instrument information).
- Creating and selecting the thermal method on the controller.
- Attaching and setting up external accessories as required (*e.g.*, purge gas, LNCA).
- Starting the experiment.

Preparing Samples

Determining Sample Size

Normally, sample weight in DSC experiments is in the range of 5 to 20 milligrams. If purity determinations are to be performed, then sample sizes of 1 to 3 milligrams are recommended. Refer to Table 3.1 to guide you when selecting the sample size and heating rates for your experiment.

Table 3.1 Determining Sample Size

Type of Measurement	Sample Size (mg)	Heating Rate (°C/min)
glass transition	10 to 20	10 to 20
melting point	2 to 10	5 to 10
kinetics (Borchardt & Daniels)	5 to 10	5 to 20
kinetics (ASTM)	*	0.5 to 20
heat capacity	10 to 70	20+
purity	1 to 3	0.5 to 1
crystallinity or oxidative stability	5 to 10	5 to 10

* Mass is inversely proportional to the heating rate. Use larger masses at slower rates, smaller masses at higher rates.

\+ Except for Modulated DSC™, see Appendix C.

Physical Characteristics

When making quantitative measurements or verifying reproducibility, it is important to ensure good thermal contact between the sample and sample pan. The physical characteristics of the sample affect the quality of this contact.

When using powdered or granular samples, spread them evenly across the bottom of the pan to minimize thermal gradients. For solid samples, select the side of your sample with the flattest surface for contact with the pan. After encapsulating the sample, ensure that the pan bottom is flat. If it is not, flatten it by pressing the pan bottom on a flat surface.

NOTE: The contact between the pan and the raised sample platform on the constantan disc is as important as the contact between the sample and sample pan.

Selecting Sample Pans

DSC and PDSC samples must be in sample pans for analysis. Use the following guidelines to select a sample pan material and configuration that meets the temperature and pressure range, composition, and reactivity requirements of your experiment.

Sample Pan Material

Aluminum pans can be used in most experiments, unless the sample material reacts with aluminum or the temperature is expected to go beyond that allowable for aluminum pans (600°C). Many other sample pan materials are available for experiments with special requirements. For example, you may wish to choose a particular pan material to improve the thermal conductance to the sample. Sample pans made of platinum, copper, or gold are commonly used when the sample reacts with aluminum or has a transition in the 600 to 725°C region; sample pans made of graphite are used when alloying or other undesirable metal-sample interactions occur. The many pan materials available enable you to study a wide variety of sample materials over the temperature and pressure range of the standard and pressure DSC cells.

♦ ***CAUTION:***

The maximum operating temperature for the DSC and pressure DSC cells is 725°C (600°C in hydrogen or an oxidizing atmosphere). The maximum operating temperature for the aluminum sample pans supplied with the cell is 600°C. To operate at temperatures above 600°C, use gold, platinum, copper, or graphite (carbon) pans in a non-oxidizing atmosphere.

NOTE:

The actual maximum pressures and temperatures achievable will depend on the pressure of the purge gas and its thermal conductivity. Not all pressures and temperatures are achieved with all gases.

Table 3.2 provides guidelines for one of the most important factors in the selection of a sample pan metal: the temperature range you plan to use in the experiment.

Table 3.2
TA Instruments
DSC Sample Pan
Temperature Ranges

Sample Pan	Usable Temperature Range (°C)
aluminum	-180 to 600
copper	-180 to 725
gold	-180 to 725
platinum	-180 to 725
graphite	-180 to 725
aluminum (SFI)*	-180 to 600
aluminum [hermetic to 300 kPa (3 atm) internal pressure]	-180 to 600
alodined aluminum [hermetic to 300 kPa (3 atm) internal pressure]	-180 to 200
gold [hermetic to 600 kPa (6 atm) internal pressure]	-180 to 725

*SFI = solid fat index

Sample Pan Configuration

Once you have selected the sample pan material to be used, you must determine the appropriate sample pan configuration. Depending on the requirements of the experiment, samples can be contained in:

- Nonhermetic pans
- Hermetic pans
- Open pans (sample pans without lids).

Nonhermetic Pans

Most samples can be run in nonhermetically crimped aluminum sample pans. These pans provide better thermal contact between sample, pan, and constantan disc than open pans; reduce thermal gradients in the sample; minimize sample spills; and enable you to retain the sample for further study.

Hermetic Pans

Hermetically-sealed sample pans have the same advantages as the nonhermetic pans, plus they have an airtight seal that can resist higher internal pressures (see Table 3.2). These pans are used for studies of: volatile liquids, materials that sublime, aqueous solutions above 100°C, and materials in a self-generated atmosphere. Because of its larger mass, a hermetic pan causes a slight loss of resolution compared with a nonhermetic pan; however, only the system time constant is affected, not the calorimetric accuracy.

As a hermetically-sealed sample is heated, the evolution of gaseous products causes the container pressure to increase. The sample container gradually deforms and may eventually leak. The container deformation will have some effect on the baseline as the container area contacting the constantan disc changes.

♦ *CAUTION:*

Avoid heating to temperatures that could cause the sample container to leak. Sample leakage could damage the constantan disc. A hermetically sealed container can withstand at least 300 kPa (3 atmospheres) of internal pressure. Some seals may contain higher pressures, and suitable precautions should be taken. Gold pans can withstand 600 kPa (6 atmospheres) of internal pressure.

Open Pans

Open pans (sample pans without lids) are used when contact with the cell atmosphere or reaction of the sample with a gas is required. You can also use hermetic pans as open pans by putting a pinhole in the lid before sealing.

SFI Pans

SFI pans (so named because they were first developed for the solid fat index test) are ideal for waxy or oily substances. They contain a platform on which the substance sits, which prevents the substance from "wicking" up the sides of the pan. This maintains a constant surface area during the experiment, which is especially important in oxidative studies, in which increased surface area could result in faster oxidation.

Encapsulating the Sample

The Sample Encapsulating Press is used to seal both nonhermetic and hermetic sample pans. Refer to Table 3.3 as a general guide for selecting the encapsulating method for your experiment.

Table 3.3 Selecting an Encapsulating Method

Sample Type	Measurement	Encapsulating Pan
solid (nonvolatile)	T_g or T_m	nonhermetic, hermetic, open
	oxidative stability	SFI or open
	C_p	nonhermetic
solid (volatile)	C_p	hermetic
liquid	crystallization T_g or T_m	hermetic, SFI, or open
	C_p	hermetic
	oxidative	SFI or open
aqueous	C_p, T_m, T_g	alodined aluminum hermetic

Preparing Nonhermetic Sample Pans

Before using the Sample Encapsulating Press, ensure that it is set up for nonhermetic crimping (see Appendix A).

Practice making a few nonhermetic sample pans to become familiar with this procedure before encapsulating your samples. If you have just changed the die (from hermetic to nonhermetic), make a few sample pans to ensure that the die has been installed properly.

1. If quantitative work will be done, weigh the sample pan and lid.

NOTE: When doing quantitative work, use tweezers to handle the sample pan and lid. Touching them with your fingers could leave residue that could affect your results.

2. Place the sample in the pan. If you are using a powder or granular sample, spread it evenly in the pan.

3. Place a lid on the pan.

   - If the sample is small or thin, powder, or granular, align the lid with the pan (see Figure 3.2).

   - If the sample is large or bulky, invert the lid and place it in the pan.

NOTE: Pans used with inverted lids should not be crimped.

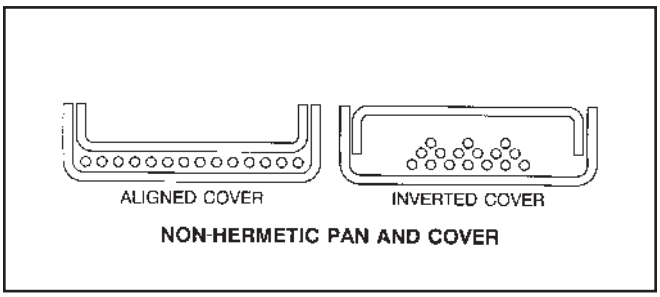


Figure 3.2 Placing the Cover Over the Pan

4. Place the sample pan in the well of the lower crimping die.

5. Pull the Sample Press lever forward until the handle hits the stop.

6. Raise the lever and remove the pan with tweezers.

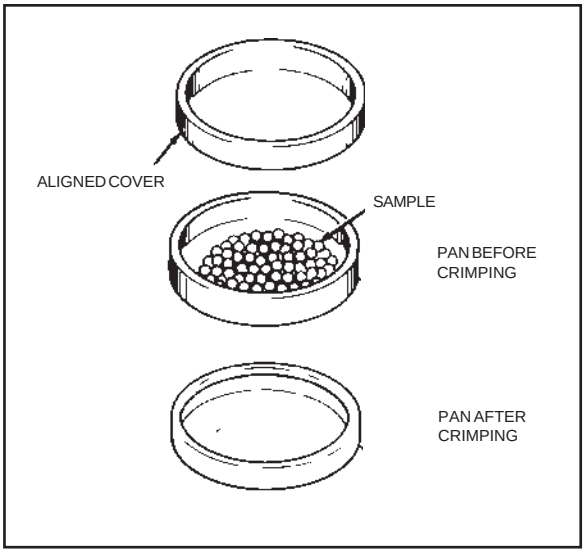


Figure 3.3 Nonhermetically Sealed Sample

7. Inspect the pan. The bottom of the pan should be smooth, and the sides should appear rolled down. If there is a ridge on the bottom of the pan, loosen the lower die holder thumbscrew and lower the bottom die holder about ¼-turn by turning it clockwise, and repeat the process from step 4. Adjust the bottom die holder until you obtain a flat pan bottom. Then, lock the bottom die holder in place by tightening the lower die holder thumbscrew.

NOTE: Large or bulky samples may rupture the pan lid. If the lid ruptures, lower the bottom die holder. Slight deformation of the lid is acceptable.

8. For quantitative work, weigh the crimped sample pan and lid (containing the sample) and determine the sample weight by subtracting the weight of the empty sample pan and lid (step 1).

9. Prepare an empty nonhermetic pan and lid (follow steps 3 through 7) for use as the reference pan.

NOTE: It is important that the same care be taken in preparing the reference pan as in preparing the sample pan. The pan bottom should be flat.

Preparing Hermetic Sample Pans

Before using the Sample Encapsulating Press, ensure that it is set up for hermetic crimping (see Appendix C).

Practice making a few hermetic sample pans to become familiar with this procedure before encapsulating your samples. If you have just changed the die (from nonhermetic to hermetic), make a few hermetic sample pans to ensure that the die has been installed properly.

To prepare a hermetic sample pan:

1. For quantitative work, weigh the sample pan and lid.

NOTE: When doing quantitative work, use tweezers to handle the sample pan and lid. Touching them with your fingers could leave residue that could affect your results.

2. Carefully place the sample in the pan. Do not allow the sample to spill onto the lip of the pan. Place the hermetic lid on the pan, and place the pan in the lower die in the Sample Press.

NOTE: When using solid samples in hermetic pans for quantitative calorimetric measurements, invert the cover to improve sample-to-pan contact and minimize dead volume. This is especially important for purity analyses.

3. Place the flat side of the preforming tool against the upper die and hold it in place. With your other hand, pull the Sample Press lever forward until the preforming tool hits the stop.

4. Raise the lever and remove the preforming tool.

5. Lower the lever again with a steady motion until the handle hits the stop. Raise the lever and remove the pan with tweezers.

6. Inspect the pan. The bottom of the pan should be smooth. There should be a smooth, complete seal around the circumference of the pan (as opposed to the rolled down appearance of a nonhermetic pan), indicating a tight seal.

7. For quantitative work, weigh the pan to determine the sample weight.

8. Prepare an empty hermetic pan and lid for use as the reference pan.

NOTE: It is important that the same care be taken in preparing the reference pan as in preparing the sample pan. The pan bottom should be flat.

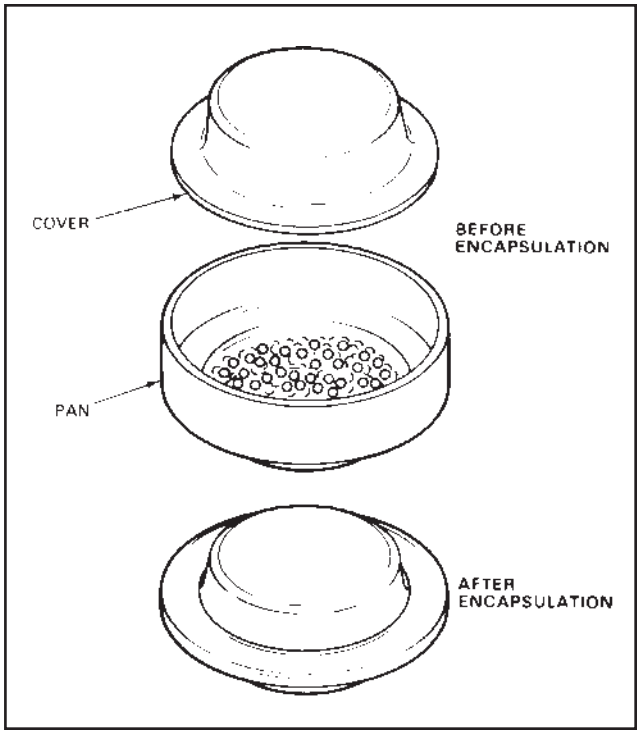


Figure 3.4 Hermetically Sealed Sample

Setting Up an Experiment

Once you have prepared the sample, the next step in your experiment is to enter the needed information in the TA controller. All of the controller functions described in this section are accessed through the Instrument Control screen. Refer to the *Thermal Solutions/Advantage User Reference Guide* to learn how to perform the following steps.

1. **Select the Instrument.**

2. **Select the Instrument Mode.**

3. **Enter Sample Information.**

4. **Enter Instrument Information.**

5. **Create and Select Thermal Methods**

   The first time you use your DSC 2910 you will need to create at least one thermal *method* to control experiments. Each method is made of several *segments*, or individual instructions (*e.g.*, Equilibrate, Ramp), that control the state of the instrument.

   For calorimetric measurements, start programming well below the onset temperature of the transition you wish to measure. This allows time for the heating rate to stabilize at the set rate and the sample and reference platforms to equilibrate. Allow at least two minutes for temperature stabilization.

♦ ***CAUTION:***

The maximum operating temperature for the DSC and pressure DSC cells is 725°C (600°C in an oxidizing atmosphere). The maximum operating temperature for the aluminum sample pans supplied with the cell is 600°C. To operate at temperatures above 600°C, use gold, platinum, copper, or graphite (carbon) pans in a non-oxidizing atmosphere.

NOTE:

The actual maximum pressures and temperatures achievable will depend on the pressure of the purge gas and its thermal conductivity. Not all pressures and temperatures are achieved with all gases.

Setting Up Accessories

If your experiment requires additional accessories, such as a purge gas, RCS, or the LNCA, ensure that they are turned on, and make any necessary adjustments before you start your experiment. Ensure that the system can achieve the temperatures in all segments of the method (*e.g.*, if subambient temperatures are required, make sure your cooling device is properly installed and filled). Use the following table as a guide in checking your DSC accessories.

Table 3.4 DSC Accessory Adjustments

External Equipment	Check or Adjustment
Air cool	Ensure that the air supply line valve from the air source is open. Ensure that the pressure is between 20 and 120 psi.
Purge gas	Make sure the correct gas is connected to the 2910 instrument. Ensure that your supply of purge gas is sufficient for the needs of the experiment. Set the purge gas flow rate.

(table continued)

Table 3.4 (continued)

External Equipment	Check/Adjustment
LNCA	Fill the LNCA tank with liquid nitrogen (see your *LNCA Operator's Manual*). Make sure the LNCA is connected to the DSC 2910. Turn on the LNCA.
NOTE:	Operation of the LNCA with the DSC 2910 is completely automatic as long as the power to the LNCA is on. The 2910 will override the LNCA controls, so there is no need to adjust them.
Refrigerated Cooling System	Install the RCS Cooling Head over the cell and turn on the RCS.
DSC Cooling Can	Install the DSC Cooling Can over the cell and fill with the desired coolant. Be ready to add more coolant as needed during the experiment.

(table continued)

Table 3.4 (continued)

External Equipment	Check/Adjustment
Gas Switching Accessory	Make sure the power switch is on. Make sure the necessary gas source(s) are properly connected.

Loading the Sample

Once the sample pan has been prepared and the sample information has been recorded, you are ready to load the sample pan into the DSC Cell.

NOTE:

If the cell has just been used, the components of the cell could be very hot. As a safe-operating practice, use the tweezers whenever handling the cell cover or silver lid.

With the DSC Cell installed on the DSC 2910, load the sample pan into the cell as follows:

1. Remove the bell jar, cell cover, and silver lid from the cell.

2. Carefully place the sample and reference pans inside the cell. Centering the pans within the grid will ensure that they are centered on the platforms (see Figure 3.5).

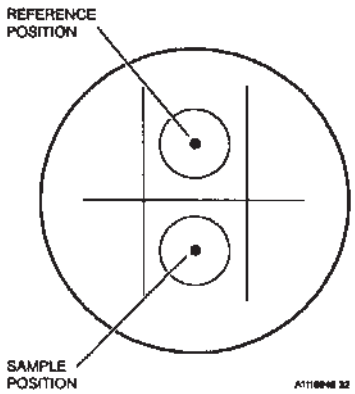


Figure 3.5
DSC Standard Cell
Pan Positions

3. Replace the silver lid, cell cover, and bell jar.

Starting an Experiment

Before you start the experiment, ensure that the DSC 2910 is online with the controller and you have entered all necessary experimental parameters.

Start the experiment by pressing the START key on the instrument keypad or **Start** on the DSC Instrument Control program, the instrument will run your method to completion.

Stopping an Experiment

If for some reason you need to discontinue the experiment, you can stop it at any point by pressing either the STOP key on the DSC 2910 keypad or **Stop** on the DSC Instrument Control program. Another function that stops the experiment is **Reject**. However, the **Reject** function discards all of the data from the experiment; the **Stop** function saves any data collected up to the point at which the experiment was stopped.

♦ ***CAUTION:*** **The REJECT function discards all experiment data.**

Important Safety Information

Please read this before using oxygen in the Pressure DSC Cell

WARNING

If excessive amounts of hydrocarbons are present in the Pressure DSC (PDSC), energetic combustion could occur causing damage to the Pressure DSC cell and possible injury to the operator. To help prevent these problems, follow these guidelines:

(1) Clean Supply Lines: The oxygen supply lines, valves, gauges, and regulators must all be free of hydrocarbons and rated for oxygen service. Check with your supplier if you are uncertain whether a component is rated for oxygen service. If the inside of the tubing smells "oily" or has liquid or a black carbon residue in it, hydrocarbons may be present. Consult with your compressed gas suppliers for a cleaning procedure.

(2) Cell Contamination: Remove the pressure housing and visually inspect the Pressure DSC cell for oil or other organic contamination. The entire oxygen pressure system must be free of hydrocarbons. If there is a possibility of hydrocarbon contamination (spilled samples, oily residue, oily smell, carbon black, etc.) in your Pressure DSC cell, immediately discontinue use. Contact TA Instruments Service at (302) 427-4050 to schedule a safety inspection, or for additional information.

WARNING

<u>Check all Supply Tubing.</u> All tubing connecting your Pressure DSC cell to other devices (oxygen cylinder, gauges, valves, regulators, etc.) should be 3.2 mm (1/8-inch) o.d. All plumbing, valves, gauges, and regulators must be rated for high pressure service to 21 MPa (3000 psig) and be free of hydrocarbons.

You should review the warnings on the previous page if you plan to use oxygen in the PDSC and any of the following conditions apply to you.

- ❑ New installation of a PDSC
- ❑ Modification of supply lines, valves or gauges
- ❑ Sample was spilled in the PDSC
- ❑ PDSC has an "oily" smell
- ❑ PDSC has not been used recently.

You may insure safe operation of your Pressure DSC if you follow the important safety instructions and warnings as directed throughout this section and the entire manual.

Important Safety Information

Please read this before using hydrogen in the special version of the Pressure DSC Cell (PN 900830.901)

!WARNING

Hydrogen gas should be used with extreme care. It is highly flammable when exposed to flame or oxidizing materials. When using hydrogen in the pressure DSC cell, the cell should be initially purged thoroughly with helium before introducing hydrogen.

At the end of the experiment, the cell should be vented into an exhaust hood and repurged with helium prior to opening the pressure container.

If you have any questions about hydrogen use, contact the U.S. TA Instruments Applications Hotline at (302) 427-4070.

Running a Pressure DSC Experiment

WARNING

Any time you open the OUT or pressure-release valve during operation, you may be applying full pressure to the external lines or components (*e.g.*, flowmeter), which may not be able to withstand full pressure. If you have a vacuum connected to the cell, the pressure would be reversed back into the cell, which may not be able to withstand an abrupt change in pressure. This could seriously damage the cell.

Experimental Procedure

Pressure DSC experiments involve the same procedures as DSC experiments, with the following exceptions:

- Loading the sample
- Purging the cell
- Controlling cell pressure
- Operating under vacuum.

WARNING

The IN and OUT valves are multi-turn needle valves, which may be damaged if excess pressure is applied to the adjustment knob. They should be closed only to the "stop" with finger-tight pressure.

Loading a Pressure DSC Sample

Once you have prepared the sample pan and entered all necessary pre-experiment data (as explained in the section on DSC experiments), you are ready to load the sample pan into the PDSC Cell. The PDSC Cell should already be installed on the DSC 2910 before you load the sample (see Chapter 2 for installation instructions).

1. Close the IN control valve (see Figure 3.7) to shut off the gas supply to the cell.

2. Slowly open the pressure-release valve and leave it open to ensure that the cell is at ambient pressure.

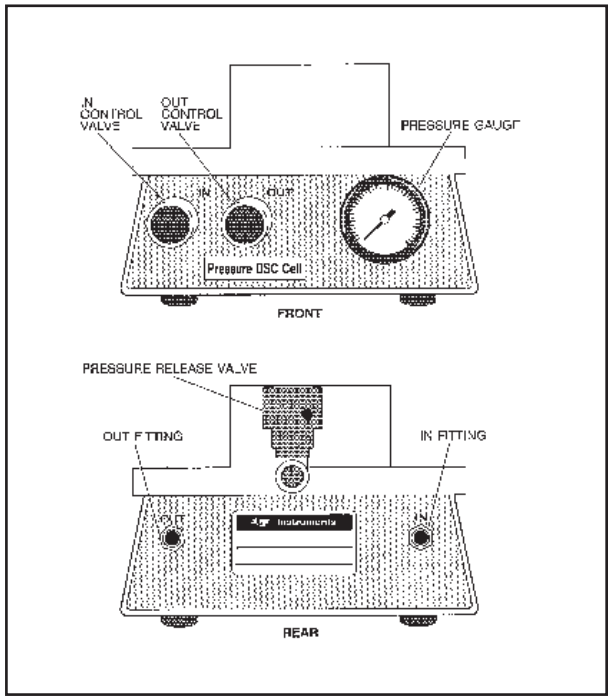


Figure 3.7 Pressure DSC Cell Controls

3. Unscrew the three thumbscrew bolts (Figure 3.8) from the top plate. Do not use tools to open or close the cell.

WARNING

If you have difficulty unscrewing the thumbscrew bolts (excessive bolt friction), you can be almost certain that the cell is still under some pressure. Recheck the valve positions as described in steps 1 and 2.

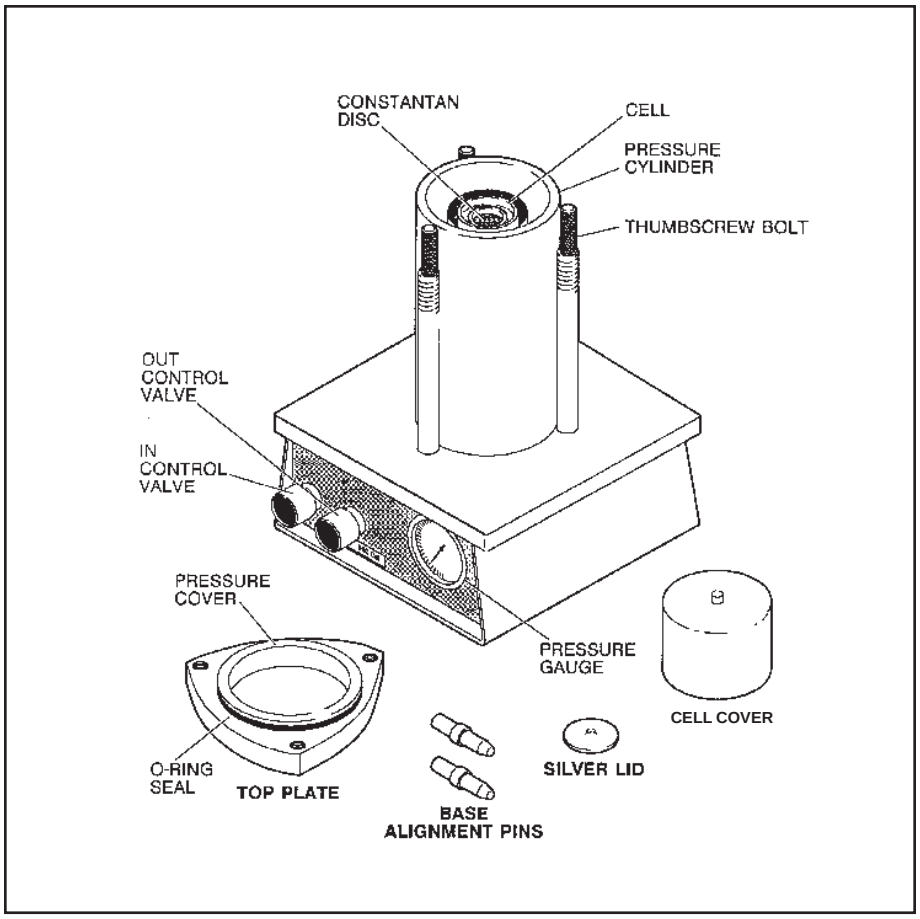


Figure 3.8
Pressure DSC Cell

4. Remove the top plate, cell cover, and silver lid.

WARNING

If the cell has just been used, these components could be very hot. As a safe operating practice, use leather gloves when handling the top plate and use tweezers whenever handling the metal cell cover or silver lid.

5. Load the sample and reference pans as you would for a standard cell. (Refer to Figure 3.5).

6. Replace the silver lid, cell cover, and top plate. Push the top plate down as far as it will go, taking care not to damage the O-ring or jar the cell, which could cause the pans to move off the dimples.

7. Uniformly *finger-tighten* the three thumbscrew bolts.

Lapping the Silver Lid and Ring

If sinusoidal baseline noise is observed in a PDSC thermal curve obtained under pressure, the silver lid and gas ring (the silver ledge on which the lid sits) may have become slightly warped and should be smoothed out with the lapping tool before the next run. The lapping tool is provided with the PDSC Cell.

1. Place the silver lid, handle side up, on a piece of fine-grit (600 grit) emery paper backed by a flat, smooth surface, and move the lid in a figure-eight motion until any deformed areas are smoothed.

2. To smooth the gas ring, attach a piece of abrasive paper (600 grit) to the lapping tool with the double-sided tape provided. Trim the paper to the size of the tool using scissors or a razor knife. Rotate the lapping tool (PN 008837.001) back and forth on the ring with light-to-moderate pressure. Clean afterward with a fiberglass brush and a light air blast.

NOTE: Be sure to wear safety glasses or goggles when cleaning the cell with air.

Gas Replacement of the PDSC Cell

If you wish to replace the gas in the PDSC Cell before your experiment, follow the guidelines in this section. Two ways to perform gas replacement are presented below, by displacement of the current gas or by evacuation of the gas present. Dynamic gas replacement during the experiment is explained under "Controlling Cell Pressure," on the next page.

WARNING

When using hydrogen as the gas [which requires a special version of the pressure DSC cell (PN 900830.901)], the cell should be initially purged thoroughly with helium before introducing hydrogen.

By Displacement

1. Close the IN control valve.
2. Open the OUT control valve.
3. Set the output regulator on the source gas cylinder to the maximum initial pressure of the experiment. If the cell is to be operated at constant volume, do not exceed 7 MPa (1000 psig).
4. Slowly open the IN control valve, and allow gas to flow through the cell for several minutes to displace ambient air.
5. Close the OUT control valve, then open the IN control valve and allow the pressure to build to the desired level.

Controlling Cell Pressure

Before you begin your experiment, make sure you have charged up the Pressure DSC to the pressure required for your experiment. Guidelines for operation at constant volume, constant pressure, and dynamic pressure are given below.

Operation at Constant Volume

After purging, check that all three cell valves are closed and that the cell is at some positive pressure.

If the cell pressure is lower than the desired starting pressure, use the IN valve to raise it. If the cell pressure is too high, use the OUT valve to lower it. However, use the IN and OUT valves conservatively; there is a lag in the reading of any pressure gauge, and if the valves are opened too rapidly or too far, the final pressure will overshoot or undershoot the desired starting pressure.

WARNING

The maximum permissible starting pressure for constant volume operation is 7 MPa (1000 psig) at room temperature. DO NOT exceed this value.

WARNING

When using hydrogen as the gas [which requires a special version of the pressure DSC cell (PN 900830.901)], the cell should be initially purged thoroughly with helium before introducing hydrogen.

Operation at Constant Pressure

For work at constant pressure, a flowmeter is required at the OUT valve to allow bleed-off of excess pressure.

WARNING

The flowmeter should have no restrictions or valves on the downstream side.

After purging, ensure that all three cell valves are closed and the cell is at some positive pressure.

1. Set the source gas regulator at the desired operating pressure.

2. Slowly open the IN valve on the cell. Wait for the internal cell pressure to stabilize at the desired operating pressure.

3. Slowly open the OUT valve to achieve 50 mL/min. Gas should vent from the cell.

Operation with Dynamic Pressure (Fixed Purge Rate)

After gas replacement, ensure that all three cell valves are closed and that the cell is at some positive pressure. Dynamic pressure operation is equivalent to operation at constant flow. An unrestricted flowmeter is required at the OUT fitting for operation in this mode.

1. Set the regulator at the source gas cylinder to an appropriate pressure.

2. Slowly open the IN valve.

3. Slowly open the OUT valve. Wait for the flow measured at the flowmeter to stabilize. If finer flow adjustment is desired, a metering flow valve may be connected between the out port and the flowmeter.

4. Adjust the OUT valve until the flowmeter indicates the desired value. If the flow rate is too low with the OUT valve fully opened, check the position of the IN valve. Carefully open the IN valve further, if necessary. If this does not raise the flow to the desired rate, the source gas pressure must be adjusted.

5. To readjust the source gas pressure, close all three valves, then repeat this procedure from step 1.

Since a flowmeter in this position is venting to the atmosphere, be sure to take the pressure differential into account when calculating flow rate over the sample at an elevated pressure.

! WARNING

Do not place any restrictions in the line from the flowmeter. A restricted line will cause the flowmeter to become pressurized.

The upper operating temperature for the Pressure DSC Cell is limited by heating rate, purge gas thermal conductivity, and test pressure.

Releasing Cell Pressure

After a PDSC run is complete, slowly release the pressure by opening the pressure-release valve.

WARNING

The exhaust gas from the pressure-release valve may be hot enough to cause burns, fires, or damage to materials.

♦ ***CAUTION:***

Rapid release of pressure can cause damage to the cell.

WARNING

When using hydrogen as the purge gas [which requires a special version of the pressure DSC cell (PN 900830.901)], the cell should be vented into an exhaust hood and repurged with helium prior to opening the pressure container.

Operating Under Vacuum

To operate the Pressure DSC under vacuum, connect a vacuum system to the pressure-release valve, and leave the two other valves closed. Procedures for cell loading and operation are the same as for standard DSC.

To maintain normal sensitivity and resolution under vacuum, you may need to use a thermally conductive material (preferably a paste) between the constantan disc and the pans. Silicone heat-sink greases (Dow Corning type 340 or equivalent) work very well. Silicone high-vacuum greases may also be used. These should not be used at temperatures over 200°C.

Running a 1600°C DTA Experiment

Experimental Procedure

1600°C DTA (Differential Thermal Analysis) experiments involve the same procedures as DSC experiments, with the following exceptions:

- Preparing samples
- Loading the sample
- Purging the cell
- Stopping the experiment.

NOTE: The COOLING GAS line is not operational when a DTA cell is installed. The Switch Air Cool function and the Air Cool option are not available.

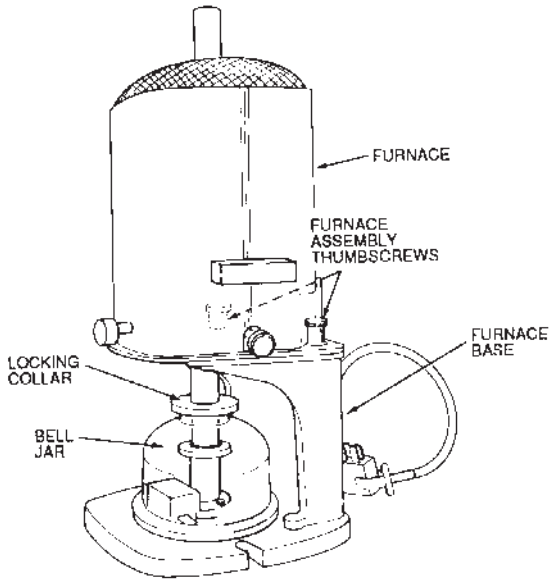


Figure 3.9
1600°C DTA Cell

Preparing Samples

Selecting Sample Cups and Liners

Platinum sample cups are available for the 1600°C DTA as macrocups (5 mm I.D., volume 75µ L). The DTA macrocups, which are used with liners, are suitable for materials that melt or sinter; the liners prevent contamination of the DTA thermocouples. The macrocups also enable you to use larger samples for increased sensitivity.

Two liner materials are available for the macrocups: alumina (ceramic) and platinum. Alumina is more porous than platinum but is otherwise sufficient for most experiments and is more economical. Advantages of the platinum liners include a slightly larger capacity due to the thinner walls and a reduced thermal gradient between the liner and the sample. The most important criterion in choosing a liner material is its reactivity with the sample; make sure the liner you choose will not amalgamate or fuse with your sample.

♦ ***CAUTION:*** **If you do not use a sample cup liner, the sample could melt and fuse with the thermocouple, resulting in the need for thermocouple replacement.**

Loading the Sample

WARNING

If the 1600°C DTA has just been used, the inside of the furnace may be extremely hot. Before loading another sample, either wait for the temperature to cool to ambient, or wear appropriate protective gloves.

1. Remove the Pyrex* cap from the furnace tube. If the furnace tube is hot, the Pyrex cap may not come loose due to differential thermal expansion of the tube and cap. Wait for the tube to cool. Do not force the cap off.

2. Loosen the two furnace assembly thumbscrews, and carefully lift the furnace off the furnace base. Lift the furnace straight up to avoid damaging the furnace tube (see Figure 3.10).

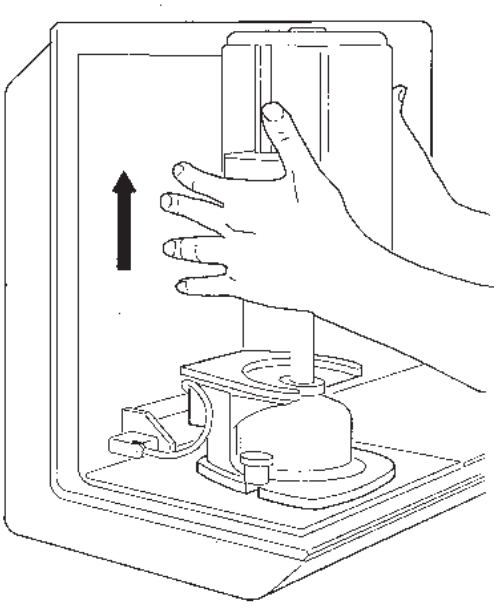


Figure 3.10 Removing the 1600°C DTA Furnace

* Pyrex is a registered trademark of Corning Glass Works.

3. Remove the two thumbscrews holding the bell jar/furnace tube in place. Then lift the whole assembly off the base.

♦ ***CAUTION:***

Be careful not to bump the furnace tube against the thermocouples while removing it. Even a slight bump could crack the ceramic sleeve of a thermocouple. If a thermocouple sleeve is cracked, the pair should be replaced.

4. Weigh and fill your sample cup. Follow the directions below when using macrocups:

   a. Using tweezers, place a macrocup on each thermocouple. Make sure the macrocups touch bottom on the posts.

   b. Weigh the sample, and then place it in a platinum or alumina liner. Place the liner in the macrocup on the left.

NOTE:

Before you weigh your sample, first make a small styrofoam* holder for the liner. Then weigh the holder and the liner, weigh the sample inside the holder/liner combination, and subtract. This will reduce spillage during weighing.

   c. Fill another liner with reference material (*e.g.*, Al_2O_3). To minimize baseline slope and offset caused by weight differences between the sample and reference, fill the reference liner to match the heat capacity of the sample.

* Styrofoam is a registered trademark of the Dow Chemical company.

d. Place the reference liner in the macrocup on the right. Ensure that the two macrocups are vertical (not slanted) and that they do not touch each other.

5. Carefully replace the bell jar/furnace tube over the thermocouple assembly and tighten the two thumbscrews to secure the cell to the instrument.

6. Carefully replace the furnace, aligning the front guide pins. Push down on the back of the furnace to seat it on the furnace base. Evenly tighten the two furnace assembly thumbscrews.

7. Look down the furnace tube and ensure that it is aligned within the furnace and is not touching the furnace.

8. Place the Pyrex cap on the furnace tube.

!WARNING

Intense heat will rise from the furnace tube if the Pyrex cap is removed while the cell is hot. Do not allow your hand, face, or any combustible material to touch or pass directly over the furnace tube.

NOTE:

If you are using purge gas, a Pyrex cap with side arm can be used. The side arm allows purging of the cell either from the base, through the cell, and out the furnace tube top, or from the top, down, and out the base. The baseline slope is not affected by the flow direction.

Differential thermal expansion (seizing) may make the Pyrex cap difficult to remove from the alumina furnace tube immediately after a run. To prevent seizing, wait for several minutes to allow the cap time to cool down before removing it.

Purging the 1600°C DTA Cell

A purge gas can be connected to either the PURGE port on the DSC 2910 or the DTA furnace cap with side arm. If you connect the purge to the furnace cap, make sure the 2910 PURGE port is open to allow the purge gas to exit.

Using the furnace cap with side arm, you can purge the cell from the bottom up (from the instrument PURGE port to the furnace cap) or from the top down (from the furnace cap to the PURGE port); the direction depends on the type of sample environment desired and the type of purge gas used. For example, if you want a pure nitrogen environment (with no air intermixed), purge from the bottom up. In general, heavy purge gases should flow from the bottom up, and light gases should flow from the top down.

If you do not use the cap with side arm when purging in the upward direction, the purge flow will be restricted and the purge gas may not be able to reach the sample.

Before you begin your experiment, set the purge gas flow rate, and ensure that your supply of purge gas is sufficient for the needs of the experiment.

NOTE: Cap the VACUUM port whenever you purge using the furnace cap with side arm. This can be done with a clamped piece of flexible tubing fitted to the port. If you use a closed furnace cap, however, closing off the VACUUM port will cause pressure build-up.

Stopping an Experiment

The procedure for stopping a DTA experiment is the same as that for a DSC experiment, with the following precautions regarding the Pyrex cap.

WARNING

Intense heat will rise from the furnace tube if the Pyrex cap is removed while the cell is hot. Do not allow your hand, face, or any combustible material to touch or pass directly over the furnace tube.

Differential thermal expansion (seizing) may make the Pyrex cap difficult to remove from the alumina furnace tube immediately after a run. Wait several minutes for the cap to cool before removing it.

Subambient Experiments

Subambient experiments can be performed with the DSC 2910 using the Liquid Nitrogen Cooling Accessory (LNCA), the Refrigerated Cooling System (RCS), and the DSC Cooling Can. Please consult the manuals that come with the LNCA and RCS for operation instructions. Instructions for operating the DSC Cooling Can are given below.

DSC Cooling Can

The DSC Cooling Can fits over the standard DSC cell. Its function is to cool the DSC cell more rapidly than the air cool function and to provide subambient operation. The reservoir is filled with coolant as needed to reach the desired temperature. An open-top bell jar, an insulation disc, and a split O-ring are also included with the accessory.

Installation instructions for the DSC Cooling Can are given in Chapter 2.

Applications

The DSC Cooling Can is used:

- To quench-cool (rapid-cool) between analyses. The DSC cell can be quench-cooled from 700°C to ambient in 3 minutes.
- To cool to a subambient temperature before a thermal program is started.

- To program-cool (by maintaining coolant level in the reservoir).

Without the insulation disc installed, the DSC Cooling Can can be used over the entire temperature range of the DSC cell.

Operation

This section contains the steps needed to operate the DSC Cooling Can.

NOTE: When the insulation disc is installed, the DSC Cooling Can should not be placed on a hot cell without coolant in the reservoir. The disc material softens at 325°C.

Quench-Cooling Between Runs

1. Carefully remove the DSC cell cover (it may be hot). Place the DSC Cooling Can (without the insulation disc) over the cell. Pour in the coolant, typically liquid nitrogen, using the open-top bell jar to minimize frost build-up on the can and DSC cell.

!WARNING **Follow the safety procedures in the front of this manual when handling liquid nitrogen.**

2. When the cell cools to ambient, remove the open top bell jar and the DSC Cooling Can, and place the sample and reference in the cell.

3. Replace the DSC Cooling Can and open top bell jar, if you want to cool the sample and reference further.

4. Remove the Cooling Can when the desired temperature is reached.

5. Replace the cell cover and glass bell jar.

6. Start a temperature ramp without using the Equilibrate segment.

NOTE: To prevent frost from forming or moisture from condensing on the constantan disc, do not remove the silver lid when the cell temperature is below ambient.

Starting a Run Below Ambient Temperature

1. Connect both the air cool and vacuum ports to one source of dry nitrogen using approximately 150 cc/min flow.

2. Place the sample and reference in the cell at ambient temperature and install the silver lid. Do not install the cell cover.

3. Place the DSC Cooling Can over the cell, and pour in the coolant using the open-top bell jar to minimize frost.

4. When the starting temperature is reached, remove the DSC Cooling Can and open top bell jar, then place the cell cover and standard bell jar over the cell. Do not remove the silver lid.

5. Wait for the sample temperature to reach a minimum.

6. Start a temperature ramp without using the Equilibrate segment.

Programmed Cooling

1. Place the sample and reference in the cell, and install the silver lid.

2. Place the insulation disc in the DSC Cooling Can to minimize baseline disturbance when the can is refilled.

NOTE: The disc material softens at 325°C. When the insulation disc is installed, the DSC Cooling Can should not be placed on a hot cell without coolant in the reservoir.

3. Place the DSC Cooling Can and open-top bell jar over the cell, and pour in the coolant.

4. Start the programmed cooling. Add coolant as needed to keep the can at least half full during programmed cooling.

NOTE: Adding coolant manually during a cooling experiment will cause variations in heat flow that are not due to the sample. These artifacts can be eliminated by using the Liquid Nitrogen Cooling Accessory or Refrigerated Cooling System whenever programmed cooling experiments are performed.

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Description of the DSC 2910

A complete DSC 2910 system includes the instrument; a standard DSC cell or Pressure DSC cell; and a controller. Both the temperatures and the heat flow associated with transitions in materials can be easily and rapidly measured by the system. The measurements provide quantitative and qualitative data relative to physical or chemical changes of a material involving endothermic (heat absorption) or exothermic (heat evolution) processes.

The optional 1600°C DTA cell can also be used with the DSC 2910. DTA measurements establish the temperature at which heat-related transitions occur, but do not provide the same accurate quantitative heat flow measurements as DSC.

The electrical connection formed between the cell and the DSC 2910 provides power to the heating element and allows transmission of thermocouple signals from the cell to the instrument. If a cell is not properly secured to the instrument, a spring-loaded interlock switch disconnects power (DSC and PDSC only).

DSC Standard Cell

The DSC cell (Figure 4.1) uses a constantan (thermoelectric) disc as a primary heat-transfer element. A silver heating block, capped with a vented silver lid, encloses the constantan disc. The selected sample and an inert reference are placed in pans that sit on raised portions of the disc. Heat is transferred through the constantan disc to both the sample and the reference pans. Differential heat flow to the sample and reference is monitored by the CHROMEL®*-constantan area thermocouples. The thermocouples are formed at the junctions of the constantan disc and the CHROMEL wafers welded to the underside of the two raised portions of the disc. CHROMEL and ALUMEL®* wires are connected to the CHROMEL wafers at the thermocouple junctions to measure sample temperature. The ALUMEL wire welded to the reference wafer is for thermal balance.

Purge gas, entering the heating block through an inlet in the DSC cell's base plate, is preheated to block temperature by circulation before entering the sample chamber through the purge gas inlet. Gas exits through the vent hole in the silver lid.

Vacuum and air cooling ports on the DSC 2910 lead to openings in the cell but not directly to the sample chamber. A bell jar, placed over the cell and sealed with an O-ring, protects the operator from evolved gases and permits cell evacuation.

* CHROMEL® and ALUMEL® are registered tradmarks of Hoskins Manufacturing Company.

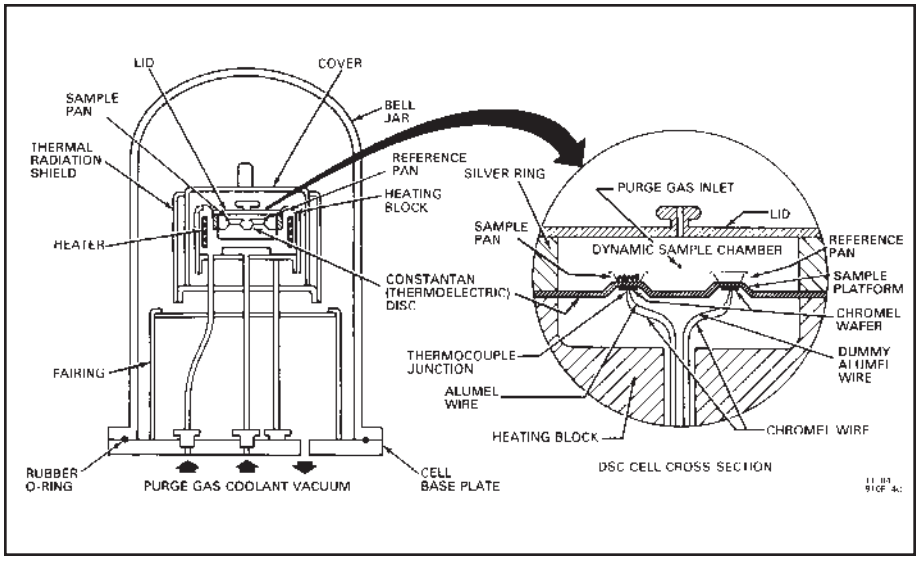


Figure 4.1
DSC Cell Cross-Section

Pressure DSC Cell

The Pressure DSC cell is the same as the standard DSC cell except that the cell is enclosed in a steel cylinder on a separate base and can be pressurized to 7 MPa (1000 psig).

1600°C DTA Cell

The 1600°C DTA Cell consists of a 1600°C furnace and furnace base, a furnace tube, a sample and reference thermocouple assembly, and sample cups. A cable for heater power and control thermocouple signals plugs into the left side receptacle on the front of the 2910 instrument.

The DTA furnace assembly is a low-mass, plug-in unit, insulated and shielded for efficient heating.

The furnace winding is a platinum alloy. The control thermocouple is platinum-platinum/13% rhodium and is positioned directly in the sidewall in a ceramic sheath.

DTA Sample and Reference Thermocouple Assembly

The 1600°C DTA thermocouple assembly consists of a matched pair of platinum-platinum/13% rhodium thermocouples inserted in ceramic tubes, a ceramic center post, and a spring clip that holds the ceramic tubes. The height of the thermocouples is critical. You can adjust the height of the thermocouples by removing the spring clip and sliding the ceramic tubes up or down the post. The thermocouple leads extend through the retainer and out the hole in the DTA furnace base, and then connect to the pin socket on the base.

DTA Sampling System

Macrocups are provided with the DTA cell on delivery. Both platinum and alumina liners are provided for the macrocups. The cups fit over the ceramic tubes, which are machined down at the ends to provide a shoulder. The macrocups with liners are suitable for materials that melt or sinter. They also enable you to use large samples for increased sensitivity.

Principles of Operation

If a sample and an inert reference are heated at a known rate in a controlled environment, the increase in sample and reference temperature will be about the same (depending on specific heat differences), unless a heat-related change takes place in the sample. If this change takes place, the sample temperature either evolves or absorbs heat. In DSC, the temperature difference between sample and reference from such a heat change is directly related to the differential heat flow.

Although the 1600°C DTA differs in configuration, particularly in how heat is coupled to the sample and reference, similar principles apply.

Cell Block Heating

The DSC 2910 controls the cell temperature by heating a silver block with a resistive wound heater and monitoring its temperature with a closely coupled control thermocouple. The appropriate amount of power supplied to the heater is determined by the difference between the temperature measured by the control thermocouple and the set point temperature (the temperature the system is attempting to reach).

Heat from the block then flows radially through the constantan disc toward the sample and reference platforms. The primary means of heat transfer to the sample and reference is through

the disc, although some heat is transferred from the lid and walls of the cell through the atmosphere.

The DSC and PDSC cells use Platinel II** control thermocouples. (The special version of the DSC 2910 designed for hydrogen experiments uses a CHROMEL®*-ALUMEL control thermocouple.) The 1600°C DTA cell uses platinum-platinum/13%rhodium.

Sample and Reference Thermocouples

The thermocouples are connected in series opposition (back-to-back) so that if the sample (T_s) and reference (T_r) temperatures are the same, the resulting electrical potential is zero. If the sample temperature is higher than the reference, the output electrical potential is one polarity; if the sample temperature is lower, the polarity is reversed.

The DSC 2910 measures the differential voltage between the thermocouples at the sample and reference platforms. This voltage is linearized/converted to mW for the DSC by the *E*-curve. The 2910 uses an average 12.3 µV/°C conversion factor for DTA.

The sample platform (the front platform) also has an ALUMEL®* lead wire forming a CHROMEL®*-ALUMEL thermocouple junction. The output from this thermocouple is monitored on the T-axis after suitable cold junction compensation.

* CHROMEL® and ALUMEL® are registered tradmarks of Hoskins Manufacturing Company.

** Platinel II is a registered trademark of Englehard Industries.

Thus, the Δq signal is determined by CHROMEL®*-constantan thermocouples, and the sample temperature is measured with a CHROMEL-ALUMEL®* thermocouple. The DSC cell baseline is very reproducible, and the cell output can be compensated to obtain a level baseline over the cell temperature range with the DSC Calibration program.

Reference junction compensation circuits are provided for Platinel—CHROMEL-ALUMEL and platinum/platinum-rhodium. The appropriate reference is connected automatically when the cell is installed.

Cell Ground

The DSC and Pressure DSC cells operate with the entire assembly (including the thermocouple) grounded through the 2910 instrument cell connector. The 1600°C DTA cell is grounded through the furnace cable.

DSC Applications

The DSC can be applied to a broad range of materials characterization, including thermal transitions in polymers:

- Glass transitions, crystallization, and melting transitions
- Curing reactions and kinetics of thermosets
- Oxidative stability of lubricants and polymers
- Purity of pharmaceuticals and organics
- Specific heat capacity of materials
- Catalyst efficiency.

Sample Types

The DSC 2910 can be used to analyze virtually any material that can be put into a DSC sample pan or DTA sample cup. The most important consideration is that the sample must make good thermal contact with the pan. Samples of solids and liquids in any of the following forms can be analyzed:

- Films
- Fibers
- Powders
- Solutions
- Composites.

Status Codes

Status codes are character strings that are continuously shown at the top left of the DSC 2910 display. These codes tell you what segment in the method is currently being performed by the instrument.

Table 4.1
Status Codes

Code	Meaning
Air Cool	The cell is being cooled by using an Air Cool segment or the **Switch Air Cool** function.
Autofill	The LNCA is being refilled from a low-pressure bulk storage tank.
Calib	The DSC 2910 is running in calibration mode.
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.

(table continued)

Table 4.1 (continued)

Code	Meaning
Complete	The thermal method has finished.
Cooling	The heater is cooling, as specified by a Ramp segment.
Equilib	The temperature is being equilibrated to the desired set point.
Err *n*	An error has occurred. The instrument display will give the error code number (*n*, a two- or three-digit code); the controller screen will also show the complete error message and provide help.
Heating	The heater temperature is increasing, as specified by a Ramp segment.
Holding	Thermal experiment conditions are holding; the program is suspended. Choose **Start** to continue the run.

(table continued)

Table 4.1 (continued)

Code	Meaning
Hot	The temperature is beyond 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 a cooling ramp being run without the 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.
Iso-track	The instrument is holding the sample at a constant temperature as specified by the Iso-track segment.
Jumping	The heater is jumping ballistically to the set point temperature.

(table continued)

Table 4.1 (continued)

Code	Meaning
No Power	No power is being measured at the heater. Check the heater switch, cell hold-down screws, and fuse.
Ready	The system has equilibrated at the initial temperature and is ready to begin the next segment. Choose **Start** 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.
Stand by	The method and method-end operations are complete.
Temp °C	The heater is in stand-by mode, and the experiment has been terminated.
Temp *	Temperature calibration is in effect. The heater is in stand-by mode, and the experiment has been terminated.

Guidelines for Quantitative Studies

You can obtain ΔH and specific heat data from DSC and PDSC experiments by following the procedures in this section. You can also calculate specific heat using the Modulated DSC™ option (refer to Appendix C for details).

Specific Heat Experiments

If you wish to calculate specific heat, follow the guidelines below when running the sample.

1. Create a baseline profile:

    a. Load the cell with empty sample and reference pans. Include lids if your experiment will use sealed pans, but do not crimp the sample pan (you will need to reuse it).

    b. Create a method that holds isothermally at the desired starting temperature for 5 minutes, heats at the desired heat rate, and then holds at the limit temperature for 2 minutes.

    c. Start the run. Deflection from the initial equilibrium point may be upward or downward, depending on the specific heat difference between the sample and reference pans.

2. Repeat the run under identical conditions with a weighed sample in the same sample pan used for the baseline profile. Do not adjust the baseline slope or perform a signal zero offset between the runs.

3. Plot the above thermograms with the data analysis program, using common limits and intervals in both plots.

4. Calculate the specific heat from the difference between the sample and blank curves at any desired temperature (see Figure 4.2).

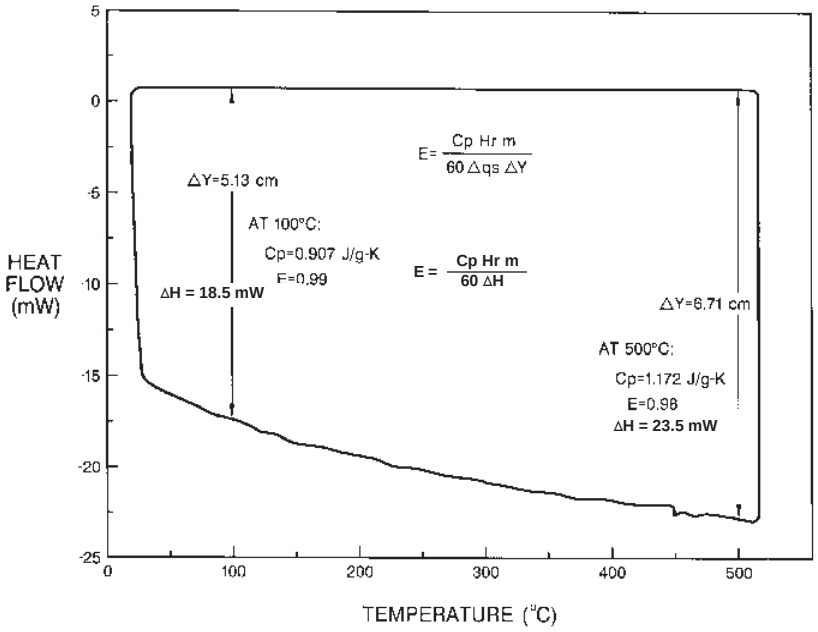


Figure 4.2
Specific Heat
of Sapphire

5. Substitute the difference into the following equation:

$$Cp = \left[\frac{60\ E}{Hr}\right]\frac{\Delta H}{m}$$

where E = cell calibration coefficient at the temperature of interest (dimensionless)

Hr = heating rate, in °C/minute

ΔH = difference in y-axis deflection between sample and blank curves at the temperature of interest, in mW

m = sample mass, in mg

C_p = specific heat, in J/g°C

The quantity $60E/Hr$ is constant under a given set of experimental conditions. It converts the y measurement directly into units of specific heat in J/g°C. For greatest accuracy, determine the value of this constant (as an entity) by running a standard material of known specific heat under conditions identical to those of the unknown sample. Then substitute the values of H, m, and C_p for the standard into the above equation at the temperature of interest.

A sapphire (Al_2O_3) standard is provided in the accessory kit for this purpose. Table 4.2 (pages 4-19 to 4-22) shows its respective specific heat values.

The values in the table were determined by Ginnings and Furukawa of the National Bureau of Standards on aluminum oxide in the form of synthetic sapphire (corundum). The sapphire pieces passed a #10 sieve but were retained by a #40 sieve, and had 99.98 to 99.99 percent purity by weight. Specific heat values below the experimental range were obtained by extrapolation of a Debye equation fitted to the experimental value at the lowest temperature.

Table 4.2
Aluminum Oxide
Specific Heat*

		Cp
°C	K	J/g°C
-183.15	90	0.0949
-173.15	100	0.1261
-163.15	110	0.1603
-153.15	120	0.1968
-143.15	130	0.2349
-133.15	140	0.2739
-123.15	150	0.3134
-113.15	160	0.3526
-103.15	170	0.3913
-93.15	180	0.4291
-83.15	190	0.4659
-73.15	200	0.5014
-63.15	210	0.5356
-53.15	220	0.5684

* Taken from D.A. Ditmars, *et.als*., J. Res. Nat. Bur. Stand., Vol 87, No. 2, pages 159-163 (1982). This is a public domain publication.

(table continued)

Table 4.2 (continued)*

°C	K	*Cp* J/g°C
-43.15	230	0.5996
-33.15	240	0.6294
-23.15	250	0.6579
-13.15	260	0.6848
-3.15	270	0.7103
0.00	273.15	0.7180
6.85	280	0.7343
16.85	290	0.7572
26.85	300	0.7788
36.85	310	0.7994
46.85	320	0.8188
56.85	330	0.8373
66.85	340	0.8548
76.85	350	0.8713
86.85	360	0.8871
96.85	370	0.9020
106.85	380	0.9161
116.85	390	0.9296
126.85	400	0.9423
136.85	410	0.9545
146.85	420	0.9660
156.85	430	0.9770
166.85	440	0.9875
176.85	450	0.9975
186.85	460	1.0070
196.85	470	1.0161
206.85	480	1.0247
216.85	490	1.0330

* Taken from D.A. Ditmars, *et.als*., J. Res. Nat. Bur. Stand., Vol 87, No. 2, pages 159-163 (1982). This is a public domain publication.

(table continued)

Table 4.2 (continued) *

°C	K	*Cp* J/g°C
226.85	500	1.0409
236.85	510	1.0484
246.85	520	1.0557
256.85	530	1.0627
266.85	540	1.0692
276.85	550	1.0756
286.85	560	1.0817
296.85	570	1.0876
306.85	580	1.0932
316.85	590	1.0987
326.85	600	1.1038
336.85	610	1.1089
346.85	620	1.1137
356.85	630	1.1183
366.85	640	1.1228
376.85	650	1.1271
386.85	660	1.1313
396.85	670	1.1353
406.85	680	1.1393
416.85	690	1.1431
426.85	700	1.1467
446.85	720	1.1538
466.85	740	1.1604
486.85	760	1.1667
506.85	780	1.1726
526.85	800	1.1783
546.85	820	1.1837
566.85	840	1.1888

* Taken from D.A. Ditmars, *et.als*., J. Res. Nat. Bur. Stand., Vol 87, No. 2, pages 159-163 (1982). This is a public domain publication.

(table continued)

Table 4.2 (continued)*

°C	*Cp* K	J/g°C
586.85	860	1.1937
606.85	880	1.1985
626.85	900	1.2030
646.85	920	1.2074
666.85	940	1.2117
686.85	960	1.2159
706.85	980	1.2198
726.85	1000	1.2237
746.85	1020	1.2275
766.85	1040	1.2312
786.85	1060	1.2348
806.85	1080	1.2383
826.85	1100	1.2417
846.85	1120	1.2451
866.85	1140	1.2484
886.85	1160	1.2516
906.85	1180	1.2548
926.85	1200	1.2578
976.85	1250	1.2653
1026.85	1300	1.2724
1076.85	1350	1.2792
1126.85	1400	1.2856
1176.85	1450	1.2917
1226.85	1500	1.2975
1276.85	1550	1.3028
1326.85	1600	1.3079
1376.85	1650	1.3128

* Taken from D.A. Ditmars, *et. als*., J. Res. Nat. Bur. Stand., Vol 87, No.2, pages 159-163 (1982). This is a public domain publication.

CHAPTER 5: Maintenance and Diagnostics

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Overview

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

WARNING

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

Routine Maintenance

Inspection

Examine the instrument periodically for good condition as follows:

- Ensure that the furnace area is clean. Any sample spillage or residue should be removed before the next experiment.
- Keep the cell connector on the DSC 2910 free of dust, debris, and moisture.

Cleaning the Instrument

You can clean the DSC 2910 keypad as often as you like. The keypad is covered with a silk-screened Mylar* overlay that is reasonably water resistant but not waterproof or resistant to strong solvents or abrasives.

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

* Mylar is a registered trademark of the Du Pont Company.

Cleaning a Contaminated Cell

A poor baseline is often the sign of a contaminated cell. DSC and PDSC cells must be cleaned properly to maintain satisfactory operation. Scraping the contamination off is not recommended because the constantan disc is very thin (about 0.1 mm, or 0.004 inches), and if the disc deforms, the baseline may be affected. Scraping can cause severe damage to the cell if it is not done carefully.

If your baseline performance begins to deteriorate, try the following recommended cleaning procedure.

- Begin cleaning by heating the cell with an air purge to 50°C above the highest operating temperature or 600°C, whichever is lower, without pans or bell jar. Use a heating rate of 20°C per minute.
- After cool-down, lightly brush out the cell with a small fiberglass eraser (included in the DSC accessory kit).
- Run the method again and compare the baselines. If there is a marked improvement but the baseline is still unacceptable, the contaminant probably oxidized and reduced to an inert ash. Run the method again and check for further improvement.
- Once the baseline is acceptable, return to normal operation.

If the constantan disc looks clean and is not bent or cracked, but the baseline problem remains, it is probably not due to contamination; the cell may need to be replaced (contact your TA Instruments service representative).

Cleaning DSC Pans

The aluminum, gold, and copper pans and the high pressure capsules provided for use with TA Instruments DSC systems are manufactured to high quality standards, including cleaning to remove contaminants that might be present from the manufacturing process. For most applications, these pans can be used as received; however, if the pans are used for high sensitivity experiments (*e.g.*, oxidative stability), an additional cleaning process is recommended before use. This procedure is taken from Appendix A of ASTM standard E1858 *Test Method for Oxidative Induction Time of Hydrocarbons by Differential Scanning Calorimeters*.

Follow the steps below to clean the TA Instruments DSC sample pans:

1. Place 200 pans in a 250 mL Erlenmeyer flask that has been fitted with a glass stopper.

2. Add approximately 150 mL of reagent grade xylene (enough to cover the pans).

3. Swirl the flask, containing the pans and xylene, for 0.5 to 2.0 min.

4. Let the flask stand for 1.0 min.

5. Decant the xylene out of the flask.

6. Repeat steps 1 through 5.

7. Add approximately 150 mL of reagent grade acetone after the second xylene wash.

8. Swirl the flask, containing the pans and acetone, for 0.5 to 2.0 min.

9. Let the flask stand for 1.0 min.

10. Decant the acetone out of the flask.

11. Repeat steps 7 through 10.

12. Rotate the flask—so that no pans adhere to the bottom or side of the flask—as you flow nitrogen at 150 to 200 mL/min over the wet pans to drive off the excess solvent. This should take approximately 5 to 6 min.

13. Return the cleaned pans to their storage container and record the date they were cleaned.

Sample Encapsulating Press

The only maintenance needed for the Sample Encapsulating Press is an occasional drop of light machine oil on the cam. Also, make sure the dies are free of material that could scratch their surfaces and impair the seal.

Diagnosing Power Problems

Fuses

The DSC 2910 contains internal fault-protection devices; however, they are not user serviceable. If any of these fail, 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 instrument's rear panel. Both slo-blo type fuses are housed in safety-approved fuse carriers, labeled F1 and F2 (see Figure 5.1). Replace these fuses with the same type and rating only.

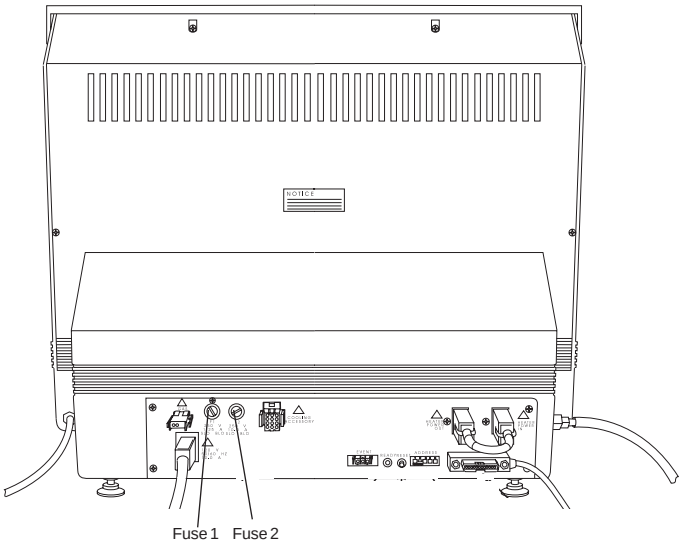


Figure 5.1
Fuse Locations

WARNING

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

Fuse F1 is in the circuit between the main electrical input and the low power loads. All power for internal operations and instrument functions, except heater power and solenoid valves, passes through this fuse. If this fuse blows, you will get no response from the instrument when you attempt to turn it on.

Fuse F2 protects the heater coils in the furnace and supplies power to the optional LNCA. Because fuse F2 does not power the internal logic, you may not know that this fuse is blown until you try to heat a sample; the 2910 passes the confidence test with this fuse open.

Fuse F2 is always checked at the beginning of a method. Power supplied by this circuit is switched by a computer-controlled relay as well as by the HEATER switch located on the DSC 2910's front panel. When both devices are active, the light in the HEATER switch will glow.

Heater Indicator Light

The indicator light in the HEATER switch on the front panel of the DSC 2910 glows whenever power is being supplied to either the heater coils in the furnace or the LNCA. This light should be on whenever a thermal method is active. If the light does not come on when the method is started, then the indicator light may be defective or a hardware problem may exist in the DSC 2910 (call your TA Instruments Service Representative).

The heater light may also remain on after a method has terminated. This can happen under the following three conditions:

1. If the DSC 2910 is configured for auto sampling and the cell furnace is being actively returned to the load temperature window after the completion of a method.

2. If an LNCA is connected to the DSC 2910 and the LNCA is switched on. (If "Air Cool" is not active, then heater power will remain on for 15 minutes after method completion to maintain operation of the jacket heater surrounding the heat exchanger. In addition, if the LNCA is autofilling, then the power will remain on until the autofill sequence is completed.)

3. If the method-end condition "Return to temperature range" function is chosen, see the *User Reference Guide* for further details.

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

- If an LNCA is connected to the DSC 2910, then STOP will terminate any active autofill operation and also turn off the heat exchanger jacket heater.

- If the instrument has an Autosampler, and the cell temperature is actively returning to the load window, then two depressions of the STOP key may be required. The first depression will abort the return to the temperature load window. The second depression will disable any active LNCA functions.

Power Failures

A power failure caused by a temporary drop in line voltage results in one of two responses by the DSC 2910:

- If the drop is fairly large and of long duration (2 seconds 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 reset. To reset, press the Reset button on the DSC 2910 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 DSC 2910 is designed for a nominal line voltage of 115 volts AC (± 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.

DSC 2910
Test Functions

The DSC 2910 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 in checking and repairing the instrument.

The Confidence Test

The DSC 2910 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 DSC 2910 system takes about 12 seconds. The longest tests are the DRAM 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 summarizes the primary confidence tests and the error codes for the DSC 2910. If any errors occur during the confidence test, call your TA Instruments service representative.

Table 5.1
DSC 2910
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*	Interface board tests
D0	Saved memory checksum

Replacement Parts

If you do not see the part you need in the table, call TA Instruments for a more complete listing.

Table 5.2
List of 2910 Parts

Part Number	Description
900155.000	1 bell jar, glass dome top for DSC Cell
900681.002	1 bell jar, glass open top for DSC Cooling Can
900660.903	1 DSC accessory kit
900610.905	1 DSC Standard Cell, new replacement
910824.001	1 DSC cleaning brush
900639.901	1 DSC cover
900394.000	1 DSC hold-down shoulder thumbscrew
911004.001	1 DSC operator's manual
911094.001	1 gasket, cell baseplate
983045.901	1 event cable
205220.021	1 fuse, 1.25 amp ceramic
205220.040	1 fuse, 10.00 amp ceramic
900014.000	1 gasket, lower cell, flat for PDSC
900410.901	1 matched pair of platinum/rhodium 1600°C DTA thermocouples
202816.339	1 O-ring, Nitrile, for bell jar
900682.001	1 O-ring, Silicon, split for DSC Cooling Can
202813.039	1 O-ring, Viton, PDSC pressure cylinder seal

(table continued)

Table 5.2 (continued)

Part Number	Description
900936.001	1 furnace tube, alumina
900786.901	200 pan bottoms, aluminum crimp
900779.901	200 pan covers, aluminum crimp
900830.985	1 PDSC Single Sample Cell, rebuilt
900843.000	1 PDSC cover
900845.000	1 PDSC thumbscrew bolt assembly
253827.000	1 power cable, 110 V
900635.000	1 silver lid for DSC, flat with hole
259538.000	1 stainless steel needle-point tweezer
202515.000	1 standard, sapphire specific heat
900902.901	1 standard, vial of indium metal
008837.001	1 gas ring lapping tool
281050.001	1 O-ring for pneumatic connections
900969.001	1 silver lid PDSC, flat
990180.000	1 pin locating/alignment for PDSC
900796.901	200 sample pans, aluminum hermetic
900793.901	200 pan covers, aluminum hermetic
900720.901	1 die, hermetic sealing
900724.901	1 set, hermetic sealing die
900719.000	1 tool, hermetic pan-preforming
900707.000	1 thermal resistor for quench cooling can

Appendix A: The Sample Encapsulating Press

Introduction

The Sample Encapsulating Press is used to seal samples in hermetic and nonhermetic sample pans. Two dies come with the press: one for hermetic sealing and one for nonhermetic sealing. This appendix explains how to change these dies.

Instructions for sealing samples with the Sample Encapsulating Press are given in Chapter 3 of this manual.

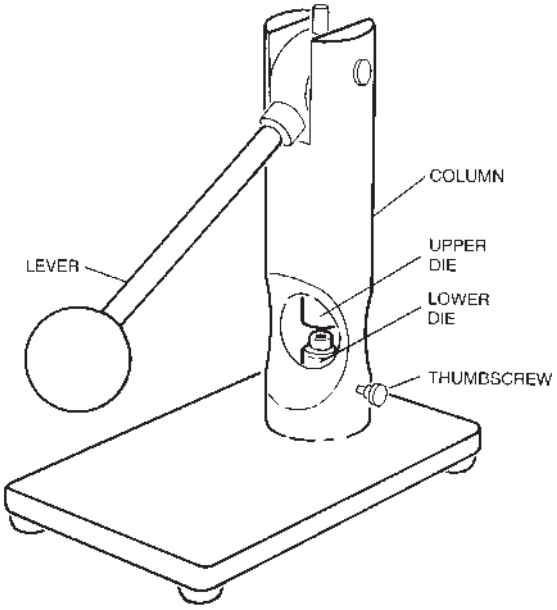


Figure A.1
Sample Encapsulating Press
With Nonhermetic Dies Installed

Setting Up the Press for Nonhermetic Sealing

The Sample Encapsulating Press is shipped with the upper nonhermetic die installed. To set up the press to make nonhermetic sample pans (when the die is set up for hermetic pans), proceed as follows:

l. Remove the hermetic die set:

   a. Loosen the thumbscrew on the column of the Sample Press (see Figure A.1).

   b. Lower the lower die holder by turning the base screw on the bottom of the press counterclockwise.

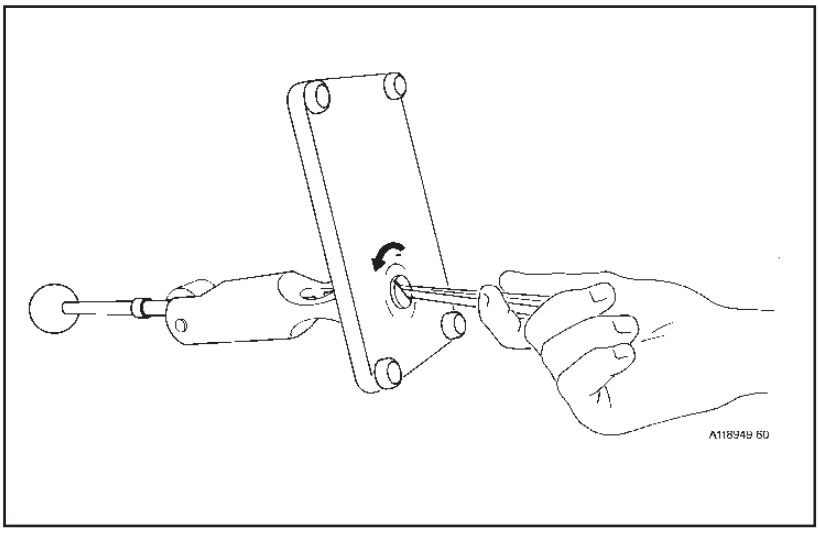


Figure A.2
Lowering the Base Screw

   c. Lift the lower hermetic die and remove it from the die holder.

2. Place the lower nonhermetic die (Figure A.3) into the lower die holder (large end up).

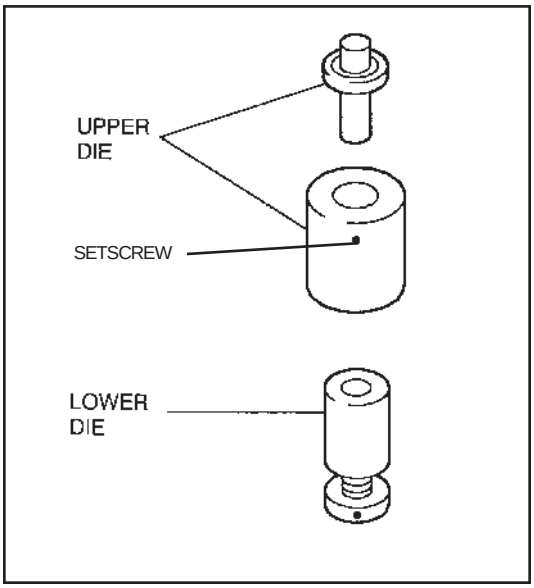


Figure A.3
The Nonhermetic Dies

3. Place the upper nonhermetic die around the plunger of the upper hermetic die (visible when the lever is lowered).

4. Push the upper nonhermetic die upward against the spring-loaded plunger and lock it in place by tightening the setscrew (Figure A.4) with a 0.050" hex wrench.

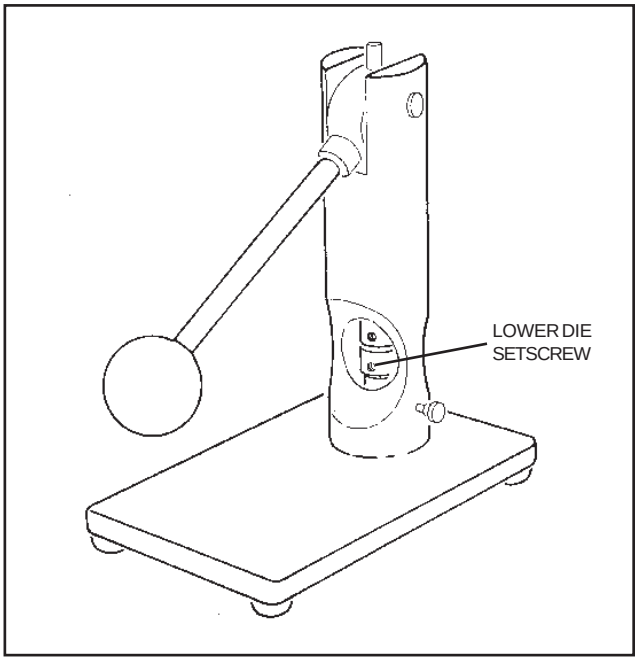


Figure A.4
Lower Die Setscrew

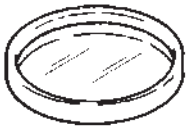
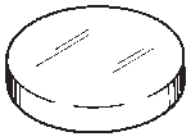
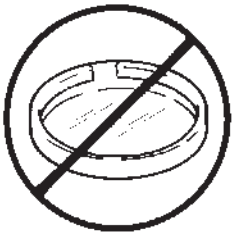
5. Adjust the height of the upper and lower dies:

   a. Pull the Sample Press lever all the way down (until it rests on the column).

   b. Turn the screw on the underside of the press clockwise as far as it will go. Then turn the screw back about ¼ turn and tighten the lower die holder thumbscrew to lock the lower die holder in place. When the press is adjusted properly, the upper and lower dies just touch. The height of the bottom die may need adjusting based on the sample height.

c. Make a few sample pans (see Chapter 3) to check the die setting. A good nonhermetic pan will have a flat bottom, and the sides of the pan will appear rolled down (see Table A.1).

Table A.1 Specifications for Nonhermetic Pans

Uniform folding of lid around circumference	
Flat surface on the bottom of the pan; no ridge around circumference	
No visible gaps between lid and pan	

Setting Up the Press for Hermetic Pans

1. Remove the nonhermetic die set:

   a. Lower the lever until you can see the setscrew on the upper nonhermetic die. If necessary, turn the upper die to access the lower die setscrew. Loosen the setscrew (Figure A.4) with a 0.050" hex wrench, raise the lever, and remove the upper die.

   b. Loosen the thumbscrew on the column of the Sample Press (see Figure A.1).

   c. Lift the lower nonhermetic die and remove it from the die holder.

2. Place the lower hermetic die (Figure A.5) into the lower die holder, either end up.

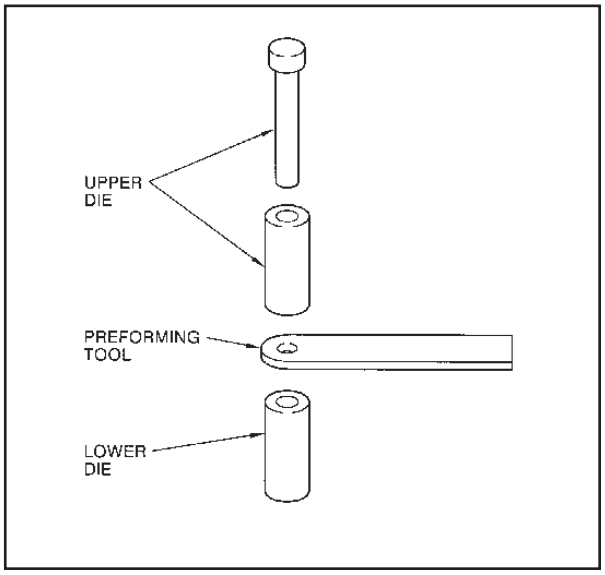


Figure A.5
The Hermetic Dies

3. Check the spring tension of the upper hermetic die (this is the die that remains in the press when the nonhermetic die is removed) by pushing up on the center plunger. If the plunger does not move, adjust the spring tension as follows:

    a. Lower the Sample Press lever. Raise the lower die holder until it contacts the upper die holder, then unscrew the holder ¼ turn. (Loosen the thumb-screw before unscrewing the lower die holder.)

    b. Keep the lever down and unscrew the upper die setscrew, letting the die come in contact with the lower die. (The upper die is spring loaded and will snap down to contact the lower die.)

    c. Tighten the setscrew on the upper die.

    d. Check the tension again. Continue to adjust until you can move the upper die plunger.

4. Adjust the setting of the upper and lower dies:

    a. Pull the lever down all the way (until it rests on the column).

    b. Turn the screw on the underside of the press clockwise as far as it will go. Then turn the screw back about ¼-turn and tighten the lower die holder thumb-screw to lock the lower die holder in place.

c. Make a few sample pans to check the die setting (see Chapter 3 for instructions). A good hermetic pan will have a flat bottom, with a complete seal around the circumference of the pan, and the sides of the pan will appear flat and smooth (see Figure A.6).

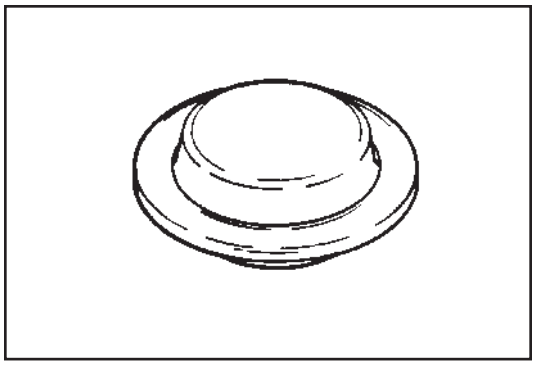


Figure A.6 Properly Sealed Hermetic Pan

Appendix B: Ordering Information

Email address: http://www.tainst.com; click on "Answerman" icon.

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: 1-302-427-4000 or 1-302-427-4040
Fax: 1-302-427-4001

HELPLINE—U.S.A.
For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis Help Desk at 1-302-427-4070.

SERVICE—U.S.A.
For instrument service and repairs, please call 1-302-427-4050.

TA Instruments Ltd.
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Fax: 61-29-8981-455

TA Instruments Italy
Division of Waters SpA
via Achille Grandi 27
20090 Vimodrone (MI), Italy
Telephone: 39-02-27421-1
Fax: 39-02-250-1827

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Introduction to Modulated DSC® *

This appendix describes how to use the Modulated DSC (MDSC) option for the DSC 2910 and DSC 2920.

MDSC is used to study the same material properties as conventional DSC including: transition temperatures, melting and crystallization, and heat capacity. However, MDSC also provides unique capabilities that increase the amount of information that can be obtained from a single DSC experiment, thereby improving the quality of interpretation. These capabilities include:

- Measurement of heat capacity and heat flow in a single experiment
- Separation of complex transitions into more easily interpreted components
- Increased sensitivity for detection of weak transitions
- Increased resolution of transitions without loss of sensitivity
- Increased accuracy in the measurement of polymer crystallinity
- Direct determination of thermal conductivity.

* Modulated DSC® and MDSC® are terms which describe proprietary technology invented by Dr. Mike Reading of ICI Paints (Slough UK) and patented by TA Instruments (U.S. Patent Nos. B1 5,224,775; 5,248,199; 5,335,993; 5,346,306).

The MDSC® option includes special enhancements to the TA controller software and the DSC 2910 and DSC 2920 software.

Although MDSC experiments can be performed with compressed air, optimum performance of the MDSC experiment often requires the use of either the Refrigerated Cooling System (RCS) or the Liquid Nitrogen Cooling Accessory (LNCA).

Option Installation

The Modulated DSC option is field installable by qualified service personnel (see separate installation procedure included with the MDSC® kit). The following are required:

- Version 1.0 or higher DSC 2920 or 2910 Software (included in kit)
- Compatible version of controller operating software
- MDSC software option key (included in kit)

Installation of the LNCA Heat Exchanger and Refrigerated Cooling System (RCS) are covered in their respective manuals.

A DSC instrument with MDSC capability properly installed can be identified by the "MDSC Installed" message on the instrument display screen following the confidence test, and by the letters "MDSC" in the instrument identification string on the configuration screen of the controller (*e.g.*, "2910 MDSC V1.0A").

The MDSC option is not compatible with the DSC 910, the DSDSC 912, the DSC 10 instrument, the 1090 controller, or the 9900 controller.

Choosing When to Use MDSC® vs. DSC

Background

Traditional DSC is a well-accepted technique for analyzing thermal transitions in materials. It provides information on the temperature at which transitions occur as well as quantitative measurement of the heat associated with the event. MDSC is an extension of DSC that provides the same information as DSC *plus* new information that permits unique insight into the structure and behavior of materials.

The need for extending the capabilities of traditional DSC, *via* MDSC, is obvious from a review of the limitations of traditional DSC. MDSC overcomes all of these limitations and is therefore the technique of choice when they are observed in traditional DSC experiments.

Limitations of Traditional DSC

Problems associated with DSC measurements fall into three general categories. In order of importance these are: analysis of complex transitions; need for increased sensitivity; and need for increased resolution.

Analysis of Complex Transitions

Most transitions are complex due to the fact that they involve multiple processes. Examples would include the enthalpic relaxation that occurs at the glass transition, and crystallization of amorphous or metastable crystalline structures prior to or during melting. Enthalpic relaxation is an endothermic process that can vary in magnitude depending on the thermal history of the material. Under some circumstances it can make the glass transition appear to be a melting transition. Simultaneous crystallization and melting make it nearly impossible to determine the real crystallinity of the sample prior to the DSC experiment. These problems are compounded further when analyzing blends of materials.

This significant limitation in traditional DSC is due to the fact that DSC measures only the sum of all thermal events in the sample. When multiple transitions occur in the same temperature range, results are often confusing and misinterpreted. MDSC® eliminates this problem by separating the total heat flow signal into its heat capacity and kinetic components. This is discussed later in this appendix.

Need for Increased Sensitivity

The ability of DSC to detect weak transitions is dependent on both short-term (seconds) noise in the heat flow signal and long-term (minutes) variations in the shape of the heat flow baseline. However, since short-term noise can be effectively eliminated by signal averaging, the real limitation for reproducibly detecting weak transitions is variation in baseline linearity. Because of the need to use different materials in the construction of DSC cells and because of changes in the thermal properties of these materials and the purge gas, all commercial DSC instruments have varying degrees of baseline camber.

MDSC® eliminates this problem by using the ratio of two signals to calculate real changes in the sample heat capacity rather than just the absolute value of the heat flow signal. This is further illustrated in the section on Principles of Operation.

Need for Increased Resolution

High resolution, or the ability to separate transitions that are only a few degrees apart requires the use of small samples and low heating rates. However, the size of the heat flow signal decreases with reduced sample size and heating rate. This means that any improvement in resolution results in a reduction in sensitivity and *vice versa*. DSC results are always a compromise between sensitivity and resolution.

MDSC® solves this problem by having effectively two heating rates. The *average heating rate* can be as low as needed to achieve the desired resolution while the *instantaneous heating rate* can be as high as needed to create a large heat flow signal.

Principles of Operation

Overview

The schematic diagram for the 2920 heat flux DSC cell is shown in Figure C.1. The sample and a reference sit on raised platforms formed in the thermoelectric (constantan) disk, which serves as the primary means of heat transfer from the temperature programmed furnace.

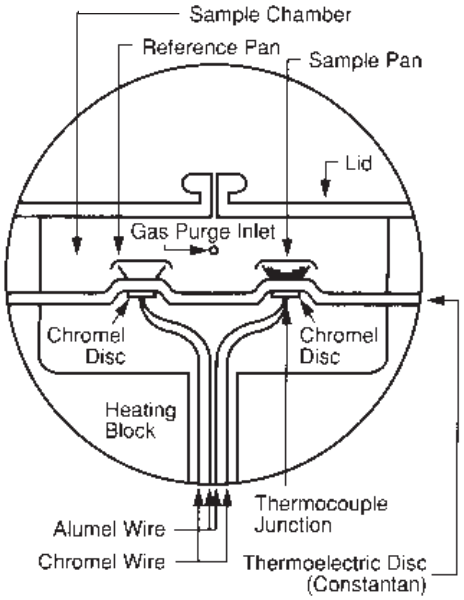


Figure C.1 Heat Flux DSC Schematic

Traditionally, the temperature of the furnace is raised or lowered in a linear fashion, and the resultant differential heat flow to the sample and reference is monitored by area thermocouples fixed to the underside of the disk platforms.

These thermocouples are connected in series and measure the differential heat flow using the thermal equivalent of Ohm's Law:

$$\frac{dQ}{dt} = \frac{\Delta T}{R_D}$$

where: dQ/dt = heat flow
ΔT = temperature difference between reference and sample
R_D = thermal resistance of constantan disc

In Modulated DSC®, the same heat flux DSC cell is used, but a sinusoidal temperature oscillation (modulation) is overlaid on the conventional linear temperature ramp, (Figure C.2). The resulting heating rate is sometimes faster than the underlying linear heating rate, and sometimes slower than the underlying rate, (Figure C.3). The actual variations in heating rate depend on three experimental variables. They are the underlying heating rate, the amplitude of modulation, and the period (frequency) of modulation.

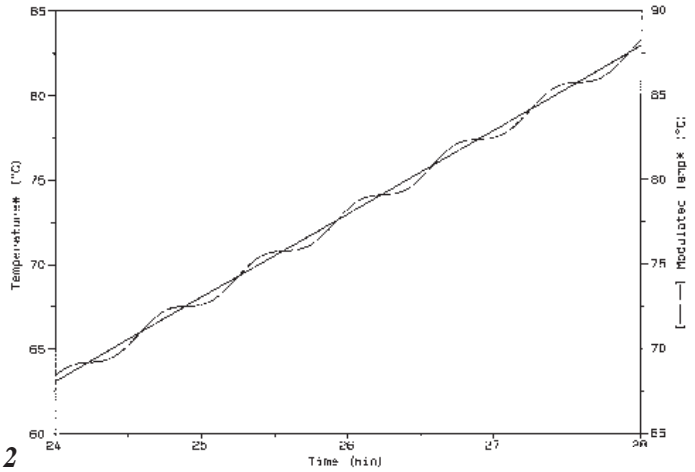


Figure C.2
Oscillation Overlaid

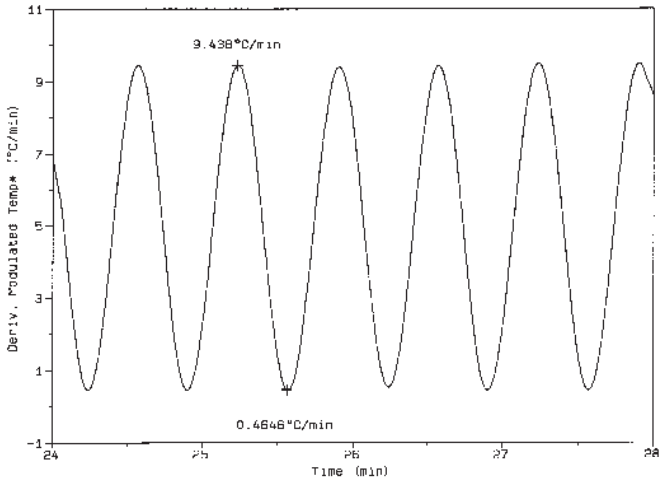


Figure C.3
Resulting Heating Rate

To appreciate the impact those variables can have on the heat flow results obtained, the general equation describing calorimetric response needs to be examined. One way to mathematically represent DSC heat flow is:

$$dQ/dt = Cp\,(dT/dt) + f(t,T)$$

where:

dQ/dt	=	heat flow
dT/dt	=	heating rate
Cp	=	sample heat capacity
t	=	time
f(t,T)	=	function of time and temperature which govern the kinetic response of any physical or chemical transition observed in DSC.

This equation shows that the total DSC heat flow is comprised of two components- one which is heating rate dependent [Cp (dT/dt)], and another which is dependent only on absolute temperature [f(t,T)]. In other words, there is one component (heat capacity component) which directly follows the modulated heating rate and one component which does not follow heating rate (kinetic component). MDSC® measures the total heat flow and separates it into these two components.

A typical "raw" MDSC experimental heat flow curve is shown in Figure C.4. The deconvoluted results are shown in Figure C.5. Deconvolution is performed in real time by Discrete Fourier Transformation software which resides in the DSC instrument.

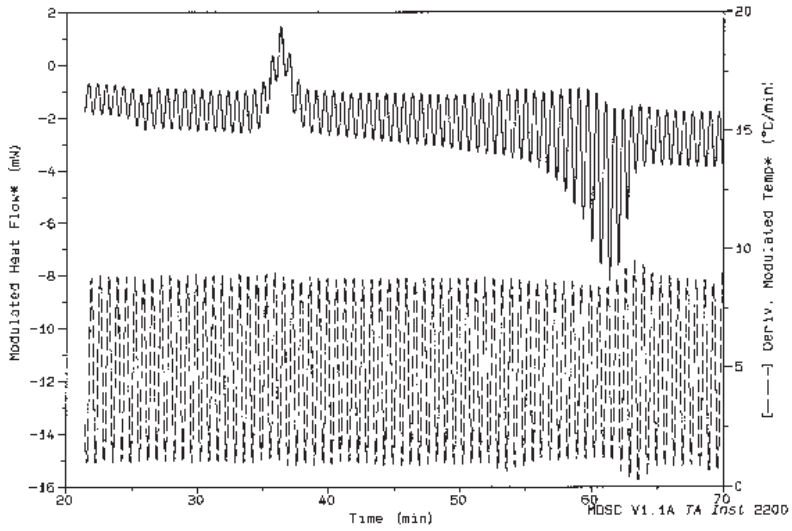


Figure C.4
Typical "Raw" MDSC
Heat Flow Curve

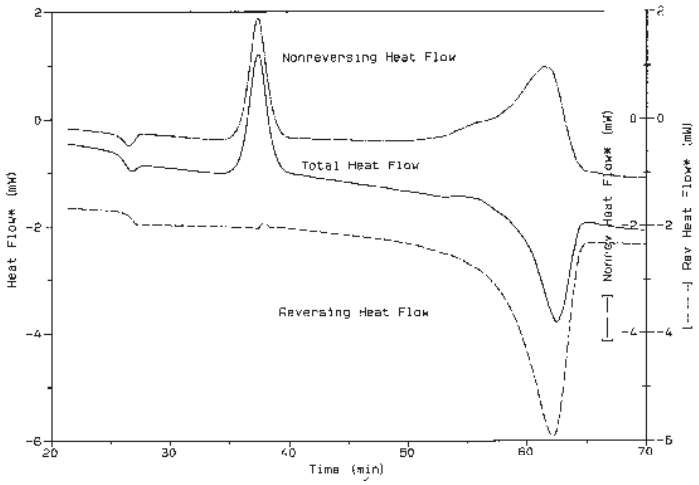


Figure C.5
Deconvoluted Results

Signal Deconvolution

How Signals are Generated

Signal deconvolution is the process of separating the raw data signals (Modulated Temperature and Modulated Heat Flow) into the average and amplitude (total change in temperature and heat flow values). In MDSC, this signal separation is accomplished by a mathematical technique known as Discrete Fourier Transformation*.

The DFT technique is used to determine the measured amplitude of the sample temperature and heat flow modulation by comparing the raw modulated data to a reference sine wave of the same frequency.

The DFT software in the DSC instrument continually measures the amplitude of the sine wave modulation in the raw sample temperature and raw heat flow signals. Using these amplitudes, the Heat Capacity signal is calculated by the following equation:

$$C_p = K_{Cp} * (Q_{amp}/T_{amp}) * (\text{Period}/2\pi)$$

*** For a description of the Discreet Fourier Transformation technique, see Press, W.H.; Flannetry, B.P.; Teukolsky, S.A.; and Vetterling, W.T., *Numerical Recipes, The Art of Scientific Computing*, 1986, Cambridge University Press, Cambridge, pp. 386-390.**

where:

C_p	=	Heat Capacity (mJ/°C)
K_{Cp}	=	Heat Capacity Calibration Constant
Q_{amp}	=	Heat Flow Amplitude (mW)
T_{amp}	=	Temperature Amplitude (°C)
Period	=	Modulation Period (sec)

Given the Heat Capacity signal (C_p), the Reversing Heat Flow is calculated by multiplying -Cp by the programmed (underlying) heating rate. The minus sign simply inverts the heat flow signal so that endothermic peaks are plotted in the downward direction. The deconvoluted Temperature and Total Heat Flow signals are computed over one complete cycle of the respective raw modulated signal. The Nonreversing Heat Flow is computed as the difference between the Total Heat Flow and the Reversing Heat Flow.

Visual Interpretation of Modulated Heat Flow

Inspection of the Modulated Heat Flow trace in Figure C.4 (see page C-15) and the resultant deconvoluted signals in Figure C.5 (see page C-16), reveal visually how the deconvolution process works.

It is evident that the Reversing Heat Flow signal (Figure C.5) is proportional to the amplitude of the heat flow oscillations (Figure C.4), and that the Nonreversing Heat Flow is proportional to the baseline shift of the oscillations. As the heat capacity of the PET sample increases through the glass transition at 25 min (75°C), the amplitude widens in Figure C.4 which results in an

increase in the Reversing Heat Flow (Figure C.5). During the recrystallization peak at 35 min (125°C), the amplitude remains essentially constant, but the baseline of the Modulated Heat Flow shifts up during the transition. Therefore, this transition does not involve a change in the heat capacity of the material, and is manifested as a peak in the Nonreversing Heat Flow.

Obtaining Multiple Heating Rate Information

It is often helpful to plot the raw Modulated Heat Flow signal (Figure C.4 on page C-15) along with the deconvoluted signals (Figure C.5). By observing the modulation "envelope" (Figure C.6), you can usually see the transitions that are observed in the deconvoluted signals.

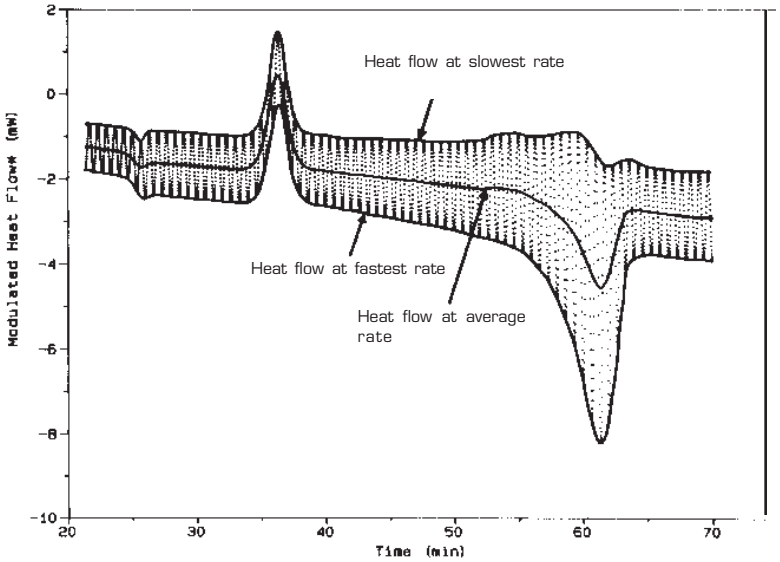


Figure C.6
Quenched Polyethylene Terephthalate Heat Flow Envelope

The envelope is defined by two boundary curves, one of which passes through all of the modulation peak maxima, and another which passes through all of the peak minima. The modulation envelope boundaries correspond to the total

DSC heat flow at the slowest and fastest heating rates during each modulation cycle. The upper boundary is the heat flow corresponding to the slowest instantaneous heating rate, which may be a slow heating rate, isothermal or even a negative heating (cooling) rate. The lower boundary corresponds to the fastest instantaneous heating rate, which normally shows the highest heat flow sensitivity. The midpoint of the envelope (the average heat flow) corresponds to the underlying or programmed ramp heating rate.

Analysis of the curves shown in Figures C.4 and C.5 near the glass transition illustrates these heating rate effects. Figure C.4 shows that the slowest heating rate during the glass transition was approximately 0.5°C/min and the fastest rate was approximately 9.5°C/min. Examination of Figure C.5 indicates that the nonreversing relaxation peak is clearly visible and is approximately the same size in all three curves. However, the baseline shift due to the change in heat capacity is virtually nonexistent in the upper (slowest rate) curve but is very pronounced in the lower (fastest rate) curve. This result is as expected since the upper curve is associated with a very slow heating rate (0.5°C/min) and the lower curve results from a relatively fast heating rate (9.5°C/min).

Just as the first derivative of a linear temperature increase corresponds to the linear heating rate, the first derivative of the Modulated Temperature (Figure C.4) corresponds to the Modulated Heating Rate. Using this signal as a guide for selecting Modulated Heat Flow data points at a constant instantaneous heating rate, it is possible to create a total heat flow curve that corresponds

to any heating rate between the slowest and fastest rates during each modulation. Transition temperatures correspond to the underlying heating rate. This approach permits the observation of heat flows at multiple heating rates from a single DSC scan. For example, if the lowest heating rate is zero degrees per minute, then the top of this signal should look like the nonreversing signal (kinetics component) because the heat capacity term goes to zero when the heating rate goes to zero.

Using MDSC®

This section describes how to use MDSC for analyzing materials. However, specific recommendations on analysis conditions for different types of transitions are covered beginning on page C-68. Before analyzing actual samples, the MDSC unit should be calibrated as described in the calibration section.

Selecting MDSC Mode and Signals

In order to use MDSC, you must select the MDSC mode of operation. This is accomplished by the following sequence of commands:

1. Select the Mode Selection window using the instrument control program.

2. Select "Modulated" mode from the drop-down list. See the *Thermal Solutions/ Advantage User Reference Guide* for further details.

Immediately after selecting the MDSC® mode, a list of possible signals will be displayed. Up to a maximum of eleven signals can be selected to be saved. The names, units and definitions for each signal are shown in Table C.1.

Table C.1
MDSC Signals

Name	Default Units	Definition
Time	min	Time since run start
Temperature*	°C	Average sample temperature
Heat Flow*	mW	Total heat flow (same as DSC)
Modulated Temperature*	°C	Measured sample temperature
Modulated Heat Flow*	mW	Measured heat flow
Reference Sine Angle*	radians	Modulation sine wave angle

(table continued)

Table C.1 (continued)

Name	Default Units	Definition
Rev Heat Flow*	mW	Deconvoluted heat capacity component of the total heat flow
Nonrev Heat Flow*	mW	Kinetic component of the total heat flow
Heat Capacity*	mJ/°C	Deconvoluted heat capacity
Temperature Amplitude*	°C	Amplitude of temperature modulation
Heat Flow Amplitude*	mW	Amplitude of the heat flow modulation

The asterisk (*) following each signal name (except Time) in Table C.1 is not intended as a footnote reference, but is actually part of the signal name. The asterisk in the name denotes that the signal is from a Modulated DSC® experiment. The asterisk was added to help distinguish MDSC experimental output signals from any other signals which may be labeled with the same or similar names.

The order of the signals shown in Table C.1 is the order in which data will be stored in the data file. If fewer than eleven signals are selected for storage, the selected *n*-signals will be shifted upwards in the table so that the stored signals

run consecutively from 1 to *n* (*e.g.*, if Nonreversing Heat Flow is not selected, then Heat Capacity will move to "Sig8/Sig-F").

At least three signals (Time, Temperature and Signal A) are always stored. Thus, the number of signals stored ranges from 3 to 11. Switching to a non-MDSC® instrument mode will change the number of signals to the standard number for that mode. Switching back to DSC Modulated mode will restore the last selection of stored MDSC signals.

In DSC Modulated mode six signals are selected by default (Time, Temperature, Modulated Temperature, Modulated Heat Flow, and Reference Sine Angle). Selecting fewer signals will reduce the size of the resultant data file. Selecting more signals will increase it. Unselected signals are not saved on disk or stored in the instrument RAM memory. Unselected signals cannot be retrieved after run completion. At first, it may seem that storing all signals all the time is the best approach. Unfortunately, doing so will result in the creation of very large data files and the rapid consumption of disk storage space.

When selecting which signals to store in the MDSC data file it is important to consider the future usage of the file. Frequently, to get maximum utility from the DSC scan, it is necessary to evaluate the data in ways that were not anticipated at the time of the experiment.

Also it is sometimes useful to go back to old data files and reanalyze them for new information. Signals that seem to be of no value initially (such as the Reference Sine Angle) may be needed for subsequent data analysis applications.

The Modulated Temperature, Modulated Heat Flow and Reference Sine Angle signals are the basic "raw" data signals from the MDSC® experiment and are required for future deconvolution of the data. If it is likely that the data will be analyzed again in the future using a new deconvolution process, then these three signals must be stored in the data file. They cannot be regenerated in post-processing of the data.

Programmed modulation amplitude and frequency are not stored in MDSC data files since these parameters can change during method execution. However, the *measured* Temperature Amplitude can be stored. If the Reference Sine Angle is stored, then the modulation period at any point in time can be computed from this signal.

For the majority of samples, there will not be a need to reanalyze the file with a new type of data analysis program. Therefore, the storage of the following signals is recommended to provide complete information in the smallest possible file:

- Time
- Temperature
- Heat Flow
- Reversing Heat Flow
- Nonreversing Heat Flow
- Heat Capacity.

The Modulate Segment

The "Modulate" segment is used to create MDSC® methods. This segment permits the entry of modulation amplitude and period (frequency) parameters for use with subsequent ramp or isothermal segments. The modulate segment will automatically appear in the method editor segment list when DSC Modulated mode is selected, and disappear when a different mode is selected. The modulate segment has the following format:

Modulate ± <amplitude>°C every <period> seconds

where:

<amplitude>	is the peak modulation temperature amplitude (0.0 to 10.0°C)
<period>	is the modulation cycle time (10.0 to 100.0 seconds)

For example:

Modulate ±0.500 °C every 40 seconds

Modulate segments execute immediately when encountered in a method, and simply set the modulation parameters to the new values provided. The last values set are used for all subsequent ramp and isothermal segments until new values are set with another modulate segment.

Modulation is not performed during set-up type segments (*i.e.*, jump, equilibrate and initial temp). If no modulate segment has been encountered in the method before a ramp or isothermal segment, then modulation will remain off. Once turned on by a modulate segment, modulation can be turned back off by inserting a modulate segment with a modulation amplitude of zero.

Normal DSC ramps and isothermal periods can be interleaved with MDSC® ramps and isothermal periods by turning the modulation on and off with the modulate segment, as described above. Note, however, that all of the selected MDSC output signals are still generated whenever the instrument is in DSC Modulated mode, even if modulation is not enabled in the method. When the modulation amplitude is set to zero, the Reversing Heat Flow, Heat Capacity, Temperature Amplitude, and Heat Flow Amplitude signals are all stored as zero. The Heat Flow, Nonreversing Heat Flow and Modulated Heat Flow are stored as conventional heat flow.

A typical MDSC method would include the following segments. Actual parameters would be selected based on transitions in the material (see page C-52 for information on selecting experimental parameters).

1. Equilibrate at 0°C
2. Modulate ±1°C every 60 seconds
3. Isothermal for 5 minutes
4. Ramp 5°C/min to 280°C.

Selecting Modulation Amplitude

The purpose of the amplitude parameter in the modulate segment is to select the magnitude of the temperature modulation sine wave. More specifically, the temperature modulation amplitude is the maximum positive or negative temperature excursion in degrees from the underlying temperature profile during one modulation cycle. The modulation amplitude can be varied from 0 to ±10°C.

The temperature modulation imposed on the underlying temperature profile will produce an accompanying modulation in the underlying heating/cooling rate. It is temperature modulation and the resultant heat flow oscillation that are deconvoluted by the MDSC® software to produce the Reversing Heat Flow, Nonreversing Heat Flow and Heat Capacity signals.

The selection of a proper temperature modulation amplitude depends on the measurement to be made. An amplitude of ±1°C is suitable for most heating, cooling or isothermal experiments. Larger amplitudes should be used when measuring very weak glass transitions and smaller amplitudes should be used for analysis of melting. The smallest recommended amplitude is ±0.1°C. Amplitudes smaller than ±0.03°C should be avoided since they are difficult to control. Specific recommended conditions for analyzing different types of transitions begin on page C-67.

Cell cooling capacity affects the ability of the instrument to achieve a selected modulation amplitude. Higher amplitudes and shorter periods require larger cell cooling capacities. (See "Cooling Devices" on page C-37 for more information on providing proper cell cooling.) A possible concern when using large amplitudes, especially at low temperatures, is that some amplitude settings cannot be achieved at some periods. In particular, shorter periods require smaller amplitude settings than do longer periods. This is a natural result of the temperature-time constant of the DSC cell.

To avoid possible distortion of the heat flow sine wave (see page C-40), it is desirable to select amplitude settings that are less than the maximum obtainable for the desired modulation period. Figure C.7 on the next page shows the maximum recommended amplitudes for 10 different modulation periods over the temperature range -150 to 500 °C when using the Liquid Nitrogen Cooling Accessory (LNCA).

When using large amplitudes it is wise to verify that a symmetric sine wave is being generated by making a trial run with empty pans and observing the Modulated Heat Flow signal. Sine wave distortion is discussed on page C-40.

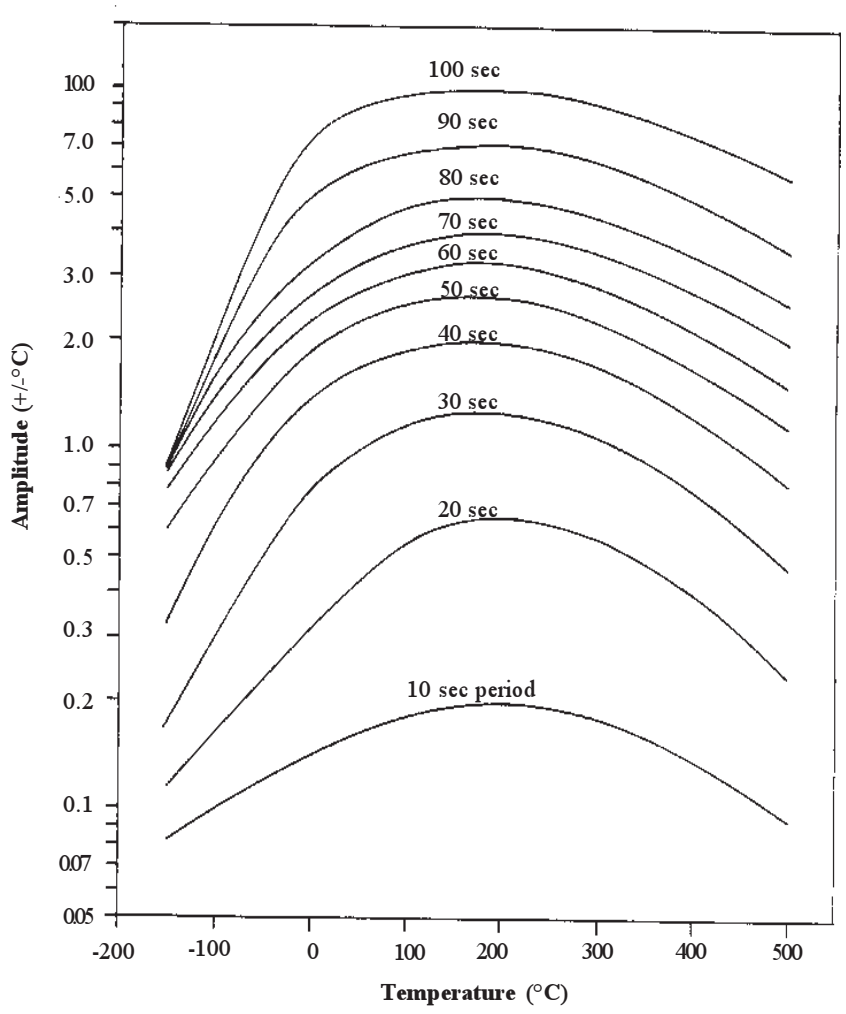


Figure C.7
Maximum Recommended Modulation Amplitudes and Periods with Liquid Nitrogen Cooling Accessory

Selecting Modulation Period

The purpose of the period parameter in the modulate segment is to select the length in time of the modulation cycle (*i.e.*, the period is the inverse of the modulation frequency). The oscillation period can be varied from 10 to 100 seconds and is automatically controlled.

The most useful period for a particular experiment depends on many factors. In general, the period should be long enough to provide for quantitative heat transfer between the sample and sensor, but short enough to permit a reasonable amount of modulation cycles during a transition. For most transitions, it is recommended that conditions be set so that a minimum of four (4) modulation cycles occur during the event. A period of 60 seconds is suggested as a starting point for initial experimentation. Specific recommended conditions for analyzing different types of transitions begin on page C-67.

The ability to achieve accurate heat capacity measurement is effected by the modulation period. Longer periods give more accurate measurements. For maximum heat capacity accuracy, a period of 80 seconds or longer is recommended.

Signal Time Delays

Modulated DSC® signals are delayed in time by 1.5 modulation cycles (1.5 times the modulation period). This delay is a natural result of the deconvolution process that must analyze data preceding and following each raw data point before the deconvoluted result can be computed. Required digital filtering of the data adds additional delay time. For this reason, MDSC data will always lag the raw instrument signals (Modulated Temperature and Modulated Heat Flow) by 1.5 cycles. Since the *x*-axis Temperature signal is a deconvoluted signal, there is no time shift between Temperature and Heat Flow.

Selecting Heating Rate

Heating rate selection in MDSC has the same effect on experimental results as in traditional DSC. Faster heating rates reduce experiment time and increase DSC sensitivity while generally sacrificing resolution. Slower rates lengthen experiment time and increase resolution at the expense of sensitivity.

In the MDSC experiment, there is an even more important effect of the heating rate. It contributes to the number of modulation cycles that occur during a transition. In order to get proper separation of the heat flow during a transition, a minimum of four (4) cycles is required. Therefore, if a transition is only 10°C wide, the heating rate should be no greater than 2.5°C/min (assuming a period of 60 seconds). The temperature width of a transition should be measured between the onset and end temperature for a glass transition and at the peak half-height for a melt or crystallization.

In practice, MDSC® can be performed at any underlying heating or cooling rate, however, rates of 5°C/min and less are recommended for most work. Use lower heating rates to improve transition resolution or to measure weak glass transitions.

Special Considerations in Creating MDSC® Methods

Ramp Start Temperature

Modulation amplitude is measured and controlled by the DSC module. Some amplitude instability will occur at the beginning of a method segment as the amplitude control stabilizes. (see Figure C.8 on the next page.) These control oscillations are not harmful to the heat flow deconvolution because the actual temperature and heat flow amplitudes are measured and used in the deconvolution calculations. Generally, these oscillations will dampen completely within 5 to 10 minutes after the start of the method segment.

When an MDSC ramp or isothermal segment starts execution the modulation amplitude is increased gradually over the first modulation period up to the specified level to prevent heater control overshoot. When this modulation ramp up is added to the 1.5-cycle deconvolution delay, plus the time for amplitude control to stabilize, the effect is that several minutes are required for the MDSC baseline to appear stable in the output data. Caution should always be used when interpreting results that are within this start-up window.

The heat flow signal of an MDSC® ramp start-up looks similar to a glass transition with a trailing relaxation peak (see Heat Flow signal in Figure C.8). All data prior to the return to stable baseline (the first 5 minutes in Figure C.8) should normally be discounted. To avoid any possibility of the amplitude stabilization affecting the quality of a transition, start the ramp at a temperature that will provide a five (5) to ten (10) minute stabilization time prior to the transition of interest.

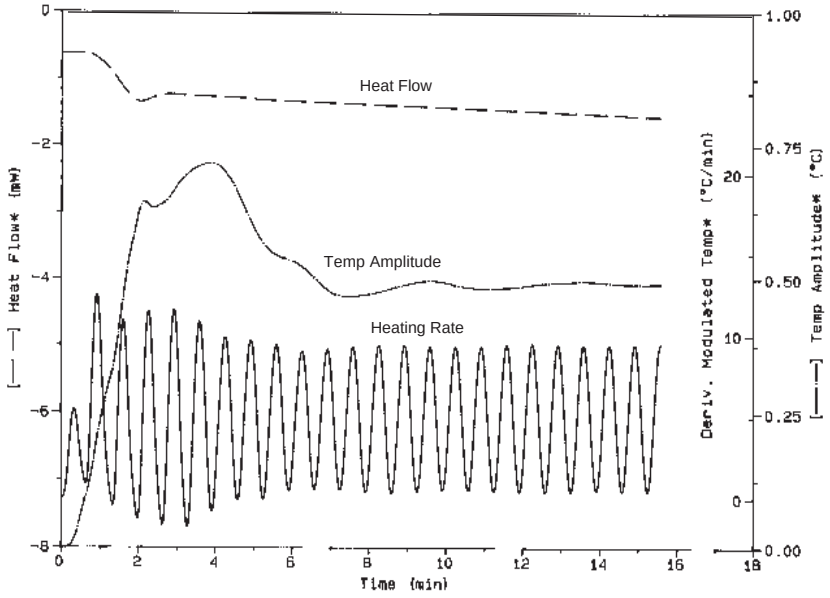


Figure C.8
Example of modulation stabilization during ramp start from ambient temperature with empty cooling can.

Ramp Final Temperature

MDSC® heat/cool ramps are controlled by the underlying heating rate, and will therefore terminate when the underlying ramp reaches the specified final temperature. Deconvoluted temperature data will appear to terminate 1.5 modulation cycles short of the final ramp temperature due to the deconvolution processing delay. This premature ramp termination can be compensated for by increasing the final temperature or by adding an isothermal segment after the ramp segment with a duration of 1.5 modulation cycles.

Isothermal MDSC

A unique feature of MDSC is the ability to perform DSC isothermal experiments that monitor changes in heat capacity, as well as endothermic and exothermic events, versus time. In this case the reversing signal will be zero due to the zero underlying heating rate. The Total Heat Flow signal (and Nonreversing signal) will contain the heat flow contribution from any nonreversing phenomenon, such as the heat flow due to a chemical reaction or decomposition. The Heat Capacity signal can be used to monitor changes in heat capacity during reactions such as thermoset cure.

Cooling

Cooling Devices

To create the relatively rapid temperature modulation, MDSC® is dependent upon cell cooling as well as cell heating. Therefore, for most experiments, a cooling device is needed. There are several alternatives available. MDSC can be used with the Liquid Nitrogen Cooling Accessory (LNCA) or the Refrigerated Cooling System (RCS). It is possible to use air cooling, but data quality will be significantly reduced. Running with the DSC cell exposed to ambient air works well above 100°C, if the ambient air temperature is not fluctuating, and if the periods are relatively long and the amplitudes small. The higher the temperature of the experiment, the larger the amplitude that can be obtained.

The Liquid Nitrogen Cooling Accessory (LNCA) can be used to obtain the widest temperature range and the largest modulation amplitude. The LNCA can be used effectively from –150°C up to 500°C. Subambient MDSC cooling rates up to 10°C/min can be achieved down to –50°C (5°C/min to –100°C). MDSC heating rates of up to 10°C/min can be achieved from –150°C up to 500°C, although rates of 5°C/min or less are more typical.

Sine Wave Distortion

Adequate cooling capacity has a large effect on the ability to achieve the selected modulation amplitude and avoid sine wave distortion. For best results, the heater power should not drop to zero watts or rise above 140 watts during any portion of the modulation cycle.

Figure C.9 shows a plot of the modulated heat flow signal from three MDSC® heating experiments at 5°C/min and a period of 40 seconds. A DSC Cooling Can without coolant was used as the cooling device. The top scan at ±1.5°C amplitude is symmetric and within the maximum recommended range for a 40-second period (See Figure C.7 on page C-31). The amplitude settings of ±3.5°C and ±5.0°C cannot be obtained under these conditions as shown by the distortion of the bottom half of each heat flow cycle in the middle and bottom scans.

As stated on page C-37, the quality of the sine wave and deconvoluted signals is greatly reduced if experiments are run with a DSC Cooling Can or compressed air as the cooling source.

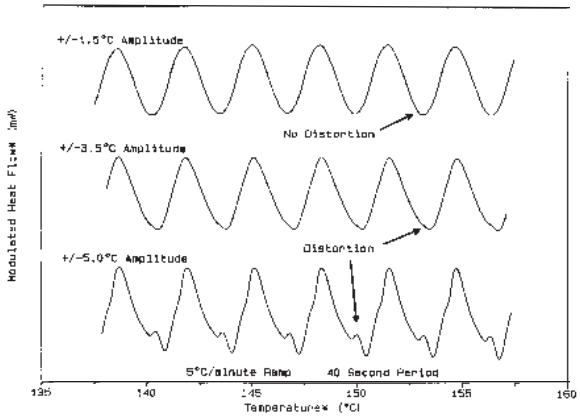


Figure C.9
Example of Heat Flow Sine Wave Distortion

When sine wave distortion occurs in the modulated heat flow signal, the resulting deconvoluted signals may be distorted, leading to misinterpretation of the data. When using previously untried combinations of period, amplitude, and cooling capacity it is wise to verify that a symmetric sine wave is being generated by making a trial run with empty pans and observing the Modulated Heat Flow signal.

Signal Display

The Signal Display window shows the MDSC® signals. Both the modulated and deconvoluted signals are shown. The following signals and units will be displayed:

- Run time (min)
- Segment time (min)
- Set point temperature (°C)
- Modulated Sig A (mW & mV) {Modulated Heat Flow}
- Offset (mV)
- Heater power (watts)
- Oscillation period (sec)
- MDSC signals 1 through 11 (appropriate units)
- Underlying dT/dt (°C/min)
- Percent memory used
- LNCA pressure.

NOTE: When Modulated mode is selected, all MDSC signals will be displayed, whether selected for output or not.

With MDSC® three different temperatures can be viewed at the same time. The temperature displayed on the instrument is the actual realtime sample temperature (as in conventional DSC). The temperature in the status line is the deconvoluted temperature (delayed by 1.5 cycles). The "Modulated Temperature" is the actual modulated sample temperature after data compression and sampling interval averaging. Therefore, during a MDSC ramp or isothermal segment, all three of these temperatures may be different.

Sampling Interval (Data Storage Rate)

Data sampling interval may be set to the same values as allowed for conventional DSC (*i.e.*, 0.2 to 1000.0 seconds/point). The default is 0.2 seconds/point (5 points/second). Increasing the sampling interval will help to reduce the size of MDSC data files. The maximum sampling interval of 5 points/second is always used to calculate the deconvoluted MDSC signals. Therefore, the accuracy of the signals is not compromised by the data storage rate. A data collection rate of 1.0 seconds/point is recommended for most MDSC experiments.

Calibrating with MDSC®

DSC Calibration

The DSC cell calibration procedure for modulated DSC is the same as for normal DSC. The calibration should be performed at the desired underlying heating rate using a conventional DSC run. Switching between standard and Modulated DSC does not require a change of calibration. If a cooling device is to be used during the experiment, then the calibration should be performed with the cooling device installed on the DSC cell.

Procedure for Measuring MDSC® Heat Capacity Calibration Constant [K(Cp)*]

An additional calibration for heat capacity is required for accurate heat capacity measurements and for proper separation of the Total Heat Flow signal into its Reversing and Nonreversing components. The heat capacity calibration is made by analyzing a sample of known heat capacity and comparing the calculated heat capacity to the literature value over the temperature range of interest. The heat capacity calibration constant is entered on the controller.

The conditions of the heat capacity calibration run should duplicate the conditions of the sample run as much as possible (*i.e.*, heating/cooling rate, modulation period and amplitude, cooling device, purge gas and flow rate, sample

pans, etc.) For optimum results, the weight of the calibration material should be chosen such that the total heat capacity of the material approximates that of the sample to be studied. Outlined on the next few pages is the recommended procedure for MDSC® heat capacity calibration.

Calibrant

Two calibration samples are provided with the MDSC Heat Capacity Calibration kit. These are each sapphire discs, cut to different dimensions but having similar weights. A different disc is used for standard pans and hermetic pans.

Modulation Conditions

The modulation conditions should be chosen so as to replicate the subsequent experimental conditions as closely as possible. Common modulation conditions include periods of 60-80 sec, and modulation amplitudes of ± 0.5°C to ±1.5°C. An underlying ramp rate of 5°C/min is sufficient, however, slower ramp rates may be used.

Temperature of Measurement

The 25 mg sapphire discs are designed as a broad-temperature range calibrant—they may be used across the entire operating range of the instrument. However, experimental precision declines as the temperature range expands. Therefore, we recommend calibrating over a 150°C range, centered in the normal operating range for your experiment. For example, if you

normally operate between 0°C and 300°C, calibrate between 75°C and 225°C. If a broader range is desired, it is possible to expand the calibration range, keeping in mind the decline in the precision of the measurement.

Calibration and Measurement Procedure

Following is a suggested procedure for heat capacity calibration. For this example, we chose:

- a range of 50°C to 200°C,
- a modulation amplitude of ±1.0°C,
- a modulation period of 60 seconds, and
- a ramp rate of 5°C/min.

1. Prepare the sample:

    a. Match the weights of the sample pan and reference pan to within 0.1 mg.
    b. Weigh the appropriate sapphire calibration disc.
    c. Record the weight.
    d. Encapsulate the disc in the pan.
    e. Crimp the lid onto the empty reference pan.

2. Create and load the following method:

    a. Equilibrate at 30°C.
    b. Modulate ±1.0°C every 60 seconds.
    c. Ramp 5°C/min to 210°C.

NOTE: A 25°C lower starting temperature is programmed to allow five minutes for modulation conditions to stabilize. A 10°C higher termination temperature is programmed to allow for the 1.5 cycle deconvolution delay.

3. Select DSC Modulated mode, and make sure the Heat Capacity signal is saved.

4. Set the MDSC® Heat Capacity Constant equal to 1.00 as follows:

   RMX Users:

   a. Select **GoTo Experimental Parameters**.
   b. Select **GoTo Module Parameters**.
   c. Enter "1.00" in "MDSC Heat Capacity Constant."

   Thermal Solutions Users:

   a. Select **Parameters** from the Main Menu.
   b. Select **Cell Calibration**.
   c. Enter "1.00" in "MDSC Heat Capacity Constant."

   Thermal Advantage Users:

   a. Select **Calibrate/Cell/Temperature Table**.
   b. Enter "1.00" in "MDSC Heat Capacity Constant."

5. Place the encapsulated sapphire disc on the sample side in the DSC cell, and the empty crimped pan on the reference side.

6. Enter the weight of the sapphire disc in Experimental Parameters, and run the loaded method.

7. When the run is finished, plot out the Heat Capacity signal versus Temperature.

8. Generate a Data Table starting at 56.85°C incrementing by 10°C.

   RMX General Analysis Users:

   a. Select **GoTo Print Report**.
   b. Select **Data Table**.
   c. Enter Start: 56.85°C
      Stop: 246.85°C
      Increment: 10°C
   d. Accept this form and send results to Printer.

   Universal Analysis Users:

   a. Select **View**.
   b. Select **Data Table**.
   c. Enter Start: 56.85°C
      Stop: 246.85°C
      Increment: 10°C
   d. Accept this form and send results to Printer.

The printer will output a data table similar to the one below:

Table C.2 Heat Capacity Example

Temperature* °C	Heat Capacity* J/g/°C
56.85	0.6850
66.85	0.7112
76.85	0.7325
86.85	0.7516
96.85	0.7694
106.85	0.7871
116.85	0.8031
126.85	0.8189
136.85	0.8328
146.85	0.8447
156.85	0.8567
166.85	0.8678
176.85	0.8771
186.85	0.8869
196.85	0.8960
206.85	0.9051
216.85	0.9154
226.85	0.9271
236.85	0.9373
246.85	0.9482

9. Compare each value of Heat Capacity to the literature value in Table C.3, Aluminum Oxide Specific Heat, beginning on the next page. (This table is also available in the Technical Reference section of the *DSC Operator's Manual*.)

Table C.3
Aluminum Oxide
Specific Heat*

		Cp
°C	K	J/g°C
–183.15	90	0.0949
–173.15	100	0.1261
–163.15	110	0.1603
–153.15	120	0.1968
–143.15	130	0.2349
–133.15	140	0.2739
–123.15	150	0.3134
–113.15	160	0.3526
–103.15	170	0.3913
–93.15	180	0.4291
–83.15	190	0.4659
–73.15	200	0.5014
–63.15	210	0.5356
–53.15	220	0.5684
–43.15	230	0.5996
–33.15	240	0.6294
–23.15	250	0.6579
–13.15	260	0.6848
–3.15	270	0.7103
0.00	273.15	0.7180
6.85	280	0.7343
16.85	290	0.7572
26.85	300	0.7788
36.85	310	0.7994
46.85	320	0.8188
56.85	330	0.8373
66.85	340	0.8548

*Taken from D.A. Ditmars, et al, *J. Res. Nat.Bur. Stand.,* Vol 87, No. 2, pages 159–163 (1982). This is a public domain publication.

(table continued)

Table C.3 Aluminum Oxide Specific Heat (continued)*

		Cp
°C	K	J/g°C
76.85	350	0.8713
86.85	360	0.8871
96.85	370	0.9020
106.85	380	0.9161
116.85	390	0.9296
126.85	400	0.9423
136.85	410	0.9545
146.85	420	0.9660
156.85	430	0.9770
166.85	440	0.9875
176.85	450	0.9975
186.85	460	1.0070
196.85	470	1.0161
206.85	480	1.0247
216.85	490	1.0330
226.85	500	1.0409
236.85	510	1.0484
246.85	520	1.0557
256.85	530	1.0627
266.85	540	1.0692
276.85	550	1.0756
286.85	560	1.0817
296.85	570	1.0876
306.85	580	1.0932
316.85	590	1.0987
326.85	600	1.1038
336.85	610	1.1089

*Taken from D.A. Ditmars, et al, *J. Res. Nat.Bur. Stand.,* Vol 87, No. 2, pages 159–163 (1982). This is a public domain publication.

(table continued)

Table C.3 (continued)*

		Cp
°C	K	J/g°C
346.85	620	1.1137
356.85	630	1.1183
366.85	640	1.1228
376.85	650	1.1271
386.85	660	1.1313
396.85	670	1.1353
406.85	680	1.1393
416.85	690	1.1431
426.85	700	1.1467
446.85	720	1.1538
466.85	740	1.1604
486.85	760	1.1667
506.85	780	1.1726
526.85	800	1.1783
546.85	820	1.1837
566.85	840	1.1888
586.85	860	1.1937
606.85	880	1.1985
626.85	900	1.2030
646.85	920	1.2074
666.85	940	1.2117
686.85	960	1.2159
706.85	980	1.2198
726.85	1000	1.2237
746.85	1020	1.2275
766.85	1040	1.2312
786.85	1060	1.2348

*Taken from D.A. Ditmars, et al, *J. Res. Nat.Bur. Stand.,* Vol 87, No. 2, pages 159–163 (1982). This is a public domain publication.

(table continued)

Table C.3 Aluminum Oxide Specific Heat (continued)*

		Cp
°C	K	J/g°C
806.85	1080	1.2383
826.85	1100	1.2417
846.85	1120	1.2451
866.85	1140	1.2484
886.85	1160	1.2516
906.85	1180	1.2548
926.85	1200	1.2578
976.85	1250	1.2653
1026.85	1300	1.2724
1076.85	1350	1.2792
1126.85	1400	1.2856
1176.85	1450	1.2917
1226.85	1500	1.2975
1276.85	1550	1.3028
1326.85	1600	1.3079
1376.85	1650	1.3128

*Taken from D.A. Ditmars, et al, *J. Res. Nat.Bur. Stand.,* Vol 87, No. 2, pages 159–163 (1982). This is a public domain publication.

10. Calculate the MDSC Heat Capacity Constant at each temperature using the following equation:

    K(Cp) = Lit. Value/Observed Value

    For example, at 56.85°C, the value of K(Cp) is calculated as follows:

    $K(Cp)^{56.85°C} = 0.8373/0.6850 = 1.22$

11. Calculate the average of all the values of K(Cp). This average value is the MDSC Heat Capacity Constant, K(Cp)*.

12. Enter the calculated value of K(Cp)* in the appropriate field.

    RMX Users:

    a. Select **GoTo Experimental Parameters**.
    b. Select **GoTo Module Parameters**.
    c. Enter value in "MDSC Heat Capacity Constant."

    Thermal Solutions Users:

    a. Select **Parameters** from the Main Menu.
    b. Select **Cell Calibration**.
    c. Enter value in "MDSC Heat Capacity Constant."

    Thermal Advantage Users:

    a. Select **Calibrate/Cell/Temperature Table**.
    b. Enter the value in "MDSC Heat Capacity Constant."

The MDSC is now calibrated for heat capacity.

Measuring Heat Capacity

To obtain accurate heat capacity measurements, the DSC cell must be calibrated for cell constant, baseline slope, temperature, and heat capacity as described in the calibration section beginning on page C-41. Heat capacity measurements are then made as follows:

1. Obtain two pans with lids that are of the same weight ±0.1 mg. Place the sample into one of the pans and crimp the lid in place. Recommended weights are as follows:

Polymers	10-15 mg
Metals	20-40 mg
Others	10-15 mg

2. Place the sample (and sample pan) to be measured on the sample side of the cell and the matching reference pan on the reference side.

3. Confirm that the Heat Capacity signal is being stored in the data file by selecting **Parameters/Mode** and checking the signals saved.

4. Create and load a method containing the same period chosen for the heat capacity calibration. Start the experiment. The calculated heat capacity will be stored in the MDSC® Heat Capacity signal.

5. Use **Universal Analysis** to observe and report the sample heat capacity at the temperature of interest.

NOTE: Long modulation periods (60 to 100 seconds) should always be used when trying to obtain maximum heat capacity accuracy. Long periods result in heat capacity calibration constants closer to unity (1.0), in contrast to much larger constants for short periods.

Guidelines for Running MDSC® Experiments

Background

MDSC is a dynamic technique that has significant advantages over traditional DSC techniques. These advantages result from the different operating principles of MDSC where the heating or cooling rate is modulated rather than held constant. Transitions (glass transition, melting, crystallization, etc.) have both kinetic and thermodynamic properties. The operating conditions of MDSC can be optimized to allow you to more easily detect and separate these properties.

In this section we will suggest "typical" operating conditions for different types of transitions. Keep in mind that these "typical" conditions are only starting points. Some materials will behave differently than others and it is up to you to further define the conditions for your particular samples.

General
Sample Preparation

The ability of MDSC® to separate overlapping transitions and to provide very high sensitivity for detection of weak transitions is totally dependent on the transfer of heat from the sample to the sensor. Therefore, some of the basic rules that apply to traditional DSC must be strictly followed. These include:

1. *Maximize the contact area between the sample and the pan.*

   To do this you must keep the sample as thin as possible in order to cover as much of the pan as possible. Do not use large irregular chunks of sample.

2. *Use lids on the DSC pans to keep the sample flat and pressed against the bottom of the pan.*

   When using hermetic pans, flatten the lid before crimping to force the sample to the bottom of the pan and to minimize its ability to move during the experiment.

3. *Use samples of 10-15 mg for polymers and keep them as thin as possible.*

   Although 10-15 mg is larger than typically used for traditional DSC experiments, it is recommended for MDSC in order to provide accurate heat capacity and to maximize the size of the total and nonreversing signals that can be smaller than typical DSC measurements. This is due to the much lower average heating rates used with MDSC.

General MDSC® Operating Parameters

Use the following general operating parameters in order to get useful results for most samples.

1. *Set up the following experimental method*:

   In order to give the entire system time to come to equilibration at the starting temperature, a 5-minute isothermal segment (as seen in step c below) is recommended.

   a. Equilibrate at start temperature
   b. Modulate ±1°C every 60 seconds
   c. Isothermal for 5 minutes
   d. Ramp 5°C/min to final temperature.

2. *Use an Amplitude of ±1°C*

   The larger the oscillation amplitude, the higher the sensitivity for detecting weak transitions. For broad, weak transitions, such as the glass transition of polypropylene or nylon fiber, it may be necessary to increase the amplitude to ±2°C. For melting transitions, small amplitudes are used so that there is no cooling (decreasing temperature) of the sample during the experiment. See Table C.4 on page C-73 for amplitude selection.

The ability to use amplitudes larger than ±2°C depends on the temperature of the experiment and on the time period of the temperature oscillation (see Figure C.7 on page C-31). In general, ±2°C provides sufficient sensitivity even for the weakest of transitions.

3. *Use an oscillation period ranging from 40 to 60 seconds.*

   For most samples, 60 seconds is the recommended period of oscillation. For narrow transitions, such as fast melts, use shorter periods. Periods of 30 seconds or less are generally not recommended. Remember to use small amplitudes when selecting short periods. For long periods it is necessary to use slower heating rates in order to achieve a minimum of four oscillations over the temperature range of the transition. Heat capacity must be calibrated at the period chosen. Use of helium purge gas, at approximately 25 mL/min, permits use of 40-second periods because the helium is more thermally conductive than nitrogen. When available, helium is the preferred purge gas for MDSC® experiments.

4. *Set the Heating or Cooling rate to 1 to 5°C/min*.

   The maximum practical heating rate is 5°C/min. (Use slower rates if you want to increase resolution.) The ideal heating rate is one that will provide a minimum of four temperature oscillations over the temperature range of the transition. For example, if

the transition being studied is 15°C wide from the extrapolated onset to the extrapolated endset, the maximum heating rate (with 60 second period) should be less than 4°C/min.

maximum rate = 1 osc/min X 15°C/4 oscillations = 3.75°C/min

To increase the number of modulation cycles during a transition, and to enhance separation, use a slower heating rate.

Example MDSC® Experiment

In this example two DSC scans of quenched polyethylene terephthalate (PET) were run in nitrogen to compare the results using conventional DSC and Modulated DSC.

Experimental Conditions

The DSC was calibrated for cell constant, baseline slope, and temperature using indium. The heat capacity calibration was performed using high density polyethylene (HDPE) as described in the section "Procedure for Measuring MDSC® Heat Capacity Calibration Constant [K(Cp)*]" on page C-41.

A sample of PET film was weighed and placed into a standard aluminum sample pan with crimped lid. A matching empty sample pan with crimped lid was used as the DSC reference. The sample was conditioned prior to each run by heating the sample to 280°C in the DSC cell and immediately quench-cooling it to room temperature by placing it on the aluminum surface of the DSC cell base. A liquid nitrogen cooling accessory (LNCA) was used for the experiment.

In the first scan (Figure C.10) a conventional heating rate of 5°C/min from ambient to 290°C was used for the method. In the second scan (Figure C.11) a modulated DSC ramp at 5°C/min from ambient to 290°C was used for the method. The modulation amplitude was ±0.53°C. The modulation period was 40 seconds. All signals were selected for data storage.

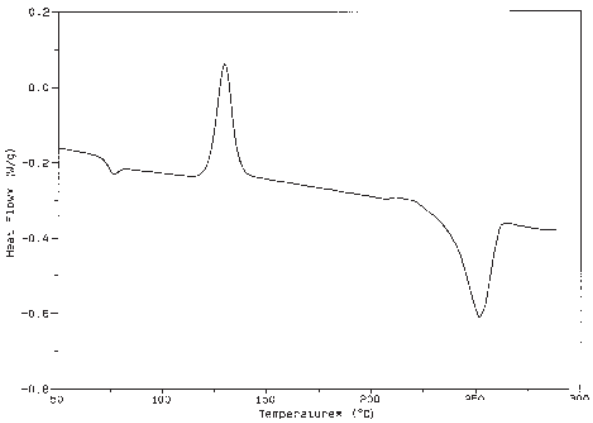


Figure C.10
PET Sample Using
Conventional Heating Rate

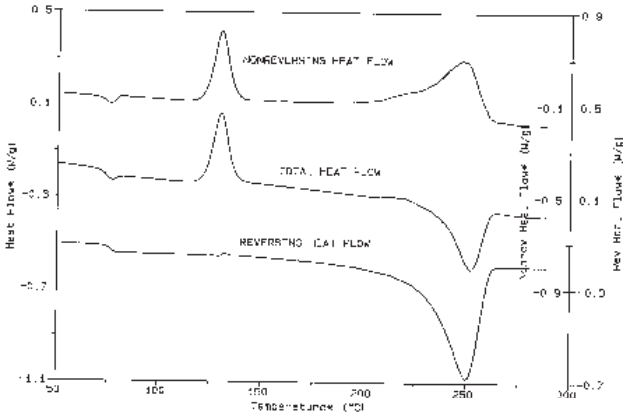


Figure C.11
PET Sample Using
a Modulated DSC
Ramp

Observations

Primarily, it is noted that the standard DSC results and the Total Heat Flow signal from MDSC® are quantitatively and qualitatively equivalent within normal experimental error.

- Glass Transition:

The glass transition at 70 °C is due to the amorphous structure in the quenched sample. The increase in molecular mobility that occurs at this temperature results in an increase in heat capacity that can be seen in the Reversing Heat Flow signal. At the same temperature, an enthalpic relaxation occurs. Since this is a kinetic process, the endothermic peak is seen in the Nonreversing Heat Flow signal.

- Cold Crystallization:

The peak observed at 125°C is due to the crystallization of the amorphous phase. Since this is a kinetic process, the peak is observed only in the Total Heat Flow and Nonreversing Heat Flow signals. A close examination of the Reversing Heat Flow signal shows a small positive shift in the baseline near 135°C due to the small decrease in the sample heat capacity as it changes from amorphous to crystalline.

- Melting:

A melting peak is present in the Total Heat Flow signal at 250°C. The irregular shape of the melt is due to an ongoing process of crystallization and crystal perfection prior to and during the melt. These processes are clearly evident in the Nonreversing Heat Flow signal. Because these

crystallization processes are exothermic, they make it impossible to detect the real onset of melting (endothermic) in the Total Heat Flow signal. The Reversing Heat Flow signal clearly shows that melting begins as low as 150°C.

Applications

The broad capability of Modulated DSC® as a valuable tool for materials research and product development is illustrated by these representative applications.

Separation of Overlapping Reversing and Nonreversing Thermal Transitions

In thermosets and semicrystalline/amorphous thermoplastics, processing can result in internal molecular stresses (thermal history effects) which are relieved on reheating. The release of these stresses sometimes appears as a small endothermic relaxation event after the glass transition. The close proximity of the endotherm to the glass transition can make interpretation difficult as shown for a B-stage epoxy (solid line) in Figure C.12 on the next page. MDSC on the other hand, separates the glass transition, which is a reversing phenomenon, from the endothermic relaxation, which is a nonreversing phenomenon. This separation greatly improves interpretation.

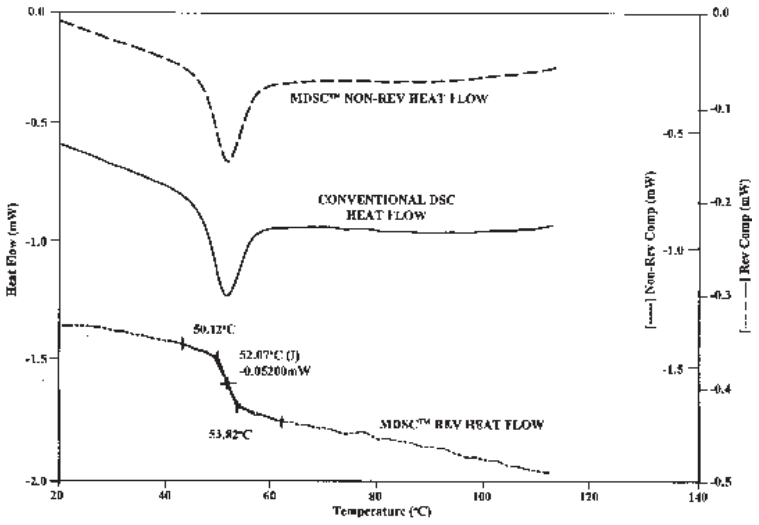


Figure C.12
B-Stage Epoxy

Traditionally, thermal history effects such as the endothermic relaxation peak are eliminated by "pretreating" the material [heating above the glass transition (T_g) and then slowly cooling] before evaluation. However, in thermosets, this type of pretreatment can advance cure and alter the results. MDSC® helps to alleviate this problem.

Figure C.13, on the next page, shows another example where MDSC improves separation and interpretation. The sample is a blend of polycarbonate (PC), polyethylene terephthalate (PET), and high-density polyethylene (HDPE). The Total Heat Flow signal shows a glass transition (T_g) near 80°C, but the large transition at 120°C is somewhat indecipherable. MDSC demonstrates that this complex thermogram actually contains two glass transitions, the PET T_g at approximately 75° and the PC T_g at approximately 145°C, as well as the HDPE melt at

approximately 120°C. These transitions involve changes in the heat capacity of the material, and are thus resolved in the Reversing Heat Flow signal. The cold crystallization of the PET occurs simultaneously with the HDPE melt, but is resolved in the Nonreversing Heat Flow signal. Thus, MDSC® is easily able to resolve complex and overlapping transitions, resulting in more accurate interpretation.

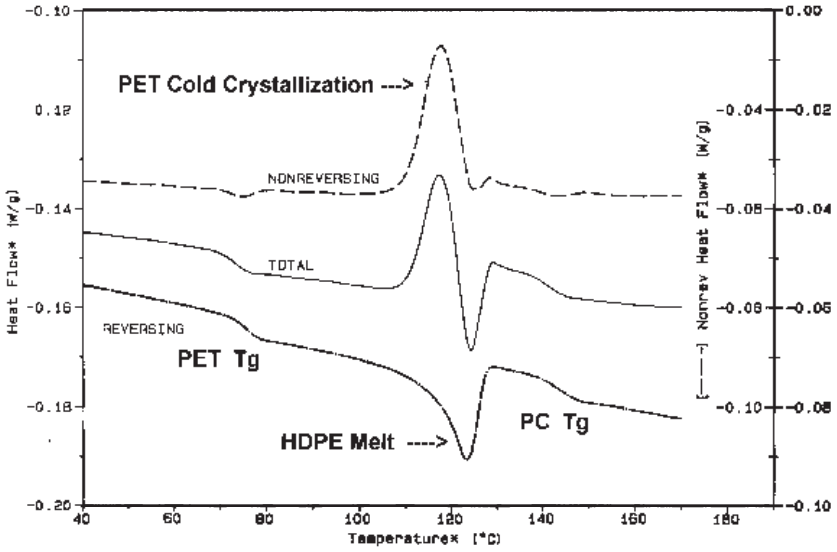


Figure C.13
Example of MDSC™
Improvement of Separation

Increased Sensitivity for Detection of Glass Transitions

Typically, glass transition (T_g) measurements in highly filled or reinforced polymers are difficult by conventional DSC. This is because T_g measurement is based on detection of a heat capacity change, and the addition of fillers and reinforces "dilutes" (weakens) the change being measured. Modulated DSC®'s high sensitivity permits the detection of a very subtle T_g.

Figure C.14 shows the MDSC results for a fiberglass reinforced composite material. The Total Heat Flow signal, which is comparable to the typical standard DSC result, exhibits a very weak, nearly non-discernible transition. The Reversing Heat Flow signal, which is based on direct heat capacity change, resolves this weak transition into a measurable T_g.

Figure C.15 illustrates the MDSC results for the glass transition of a nylon pellet with varying degrees of moisture. Moisture content can affect the temperature and intensity of the glass transition. The improved sensitivity of MDSC allows for the detection of these subtle shifts, as demonstrated in Figure C.15.

Specific recommended conditions for analyzing different types of transitions begin on page C-71.

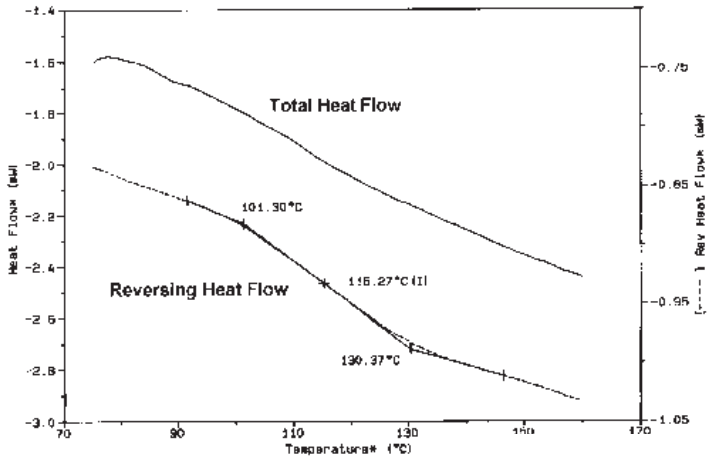


Figure C.14
Tg of Fiberglass-Reinforced Composite

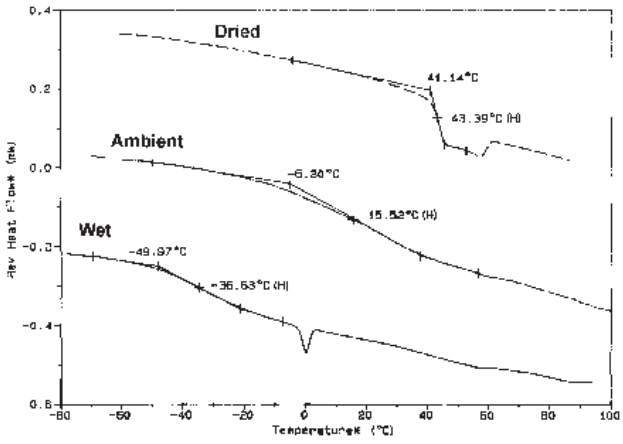


Figure C.15
Effect of Moisture on the Tg of Nylon

Direct Measurement of Heat Capacity

Heat Capacity (Cp) measurement by conventional DSC is a tedious process requiring multiple experiments and considerable operator expertise to obtain results with reasonable accuracy and precision. MDSC® provides the unique ability to measure heat capacity directly in a single experiment, even at very slow underlying heating rates.

Figure C.16 below shows the results from three separate MDSC evaluations of polystyrene. The crosses indicate reported literature Cp values at several temperatures for comparison. The glass transition is present as a step change in heat capacity at about 100°C. The table in the upper left hand corner of the figure compares the typical precision and noise, as well as the number of experiments, associated with heat capacity measurements by conventional DSC (based on ASTM round-robin results) and MDSC. MDSC provides better results in less experimental time.

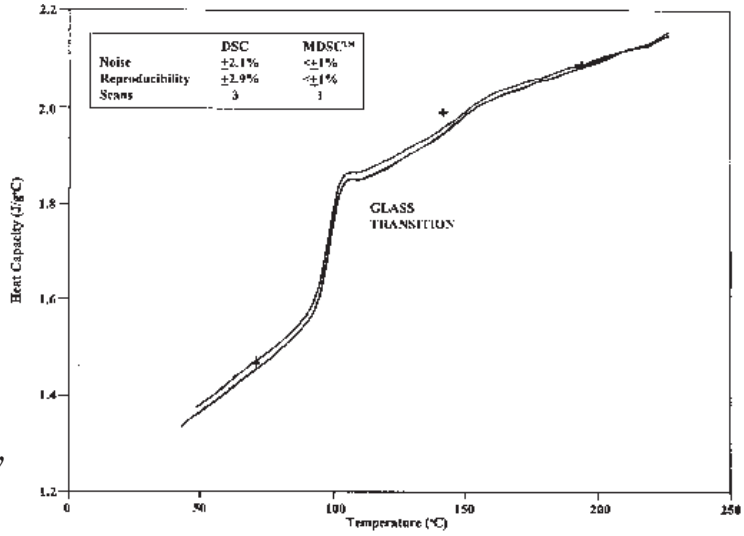


Figure C.16 Heat Capacity of Polystyrene

Isothermal Cure Evaluation

Modulated DSC® has the ability to generate an instantaneous heating rate during isothermal experiments which allows measurements to be made that are not possible in conventional DSC. The results for a high-temperature epoxy cured isothermally at 90°C (Figure C.17) illustrate this point. The solid line is the Nonreversing Heat Flow. It indicates an exothermic peak which represents the Heat of Cure equivalent to that seen by conventional DSC. The dashed line is the Heat capacity signal.

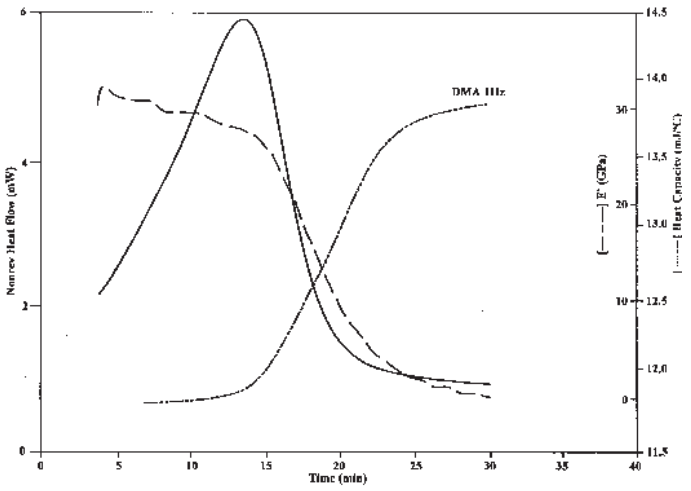


Figure C.17
Epoxy Isothermal Cure Evaluation

In theory, heat capacity should decrease as the monomer polymerizes because polymerization, or cross-linking, causes the internal molecular motion to decrease (in contrast to the heat capacity increase observed at T_g during heating in an amorphous polymer). The MDSC® heat capacity does decrease as expected. However, the onset of heat capacity decrease occurs after the exothermic peak maximum in the Nonreversing Heat Flow signal. This means that heat capacity changes more dramatically during cross-linking (the final stage of cure) than during linear polymerization (the first stage of cure). Evaluation by dynamic mechanical analysis (DMA) supports this conclusion since the storage modulus (dash-dot line) increases at the same temperature as the heat capacity begins to decrease.

Direct Measurement of Thermal Conductivity

Thermal conductivity is a measure of the ease at which heat is transmitted through a material and is a basic material property. Determination of a material's thermal conductivity is important in evaluating its utility for specific applications. In many of these applications, a textbook value or a single measurement near the temperature of use is sufficient to make a decision. In a few cases, however, the material's composition varies widely enough that regular measurement of thermal conductivity is required.

As discussed previously, MDSC® has the ability to directly measure heat capacity of a material, and can also directly monitor heat capacity changes as a function of temperature. Since heat capacity and thermal conductivity are related properties, MDSC can be used to directly measure the thermal conductivity of certain insulating materials including polymers, ceramics and glasses This is performed by measuring a sample's specific heat capacity directly *via* MDSC. The *apparent* heat capacity of a larger sample of the same material of known weight and dimension is then measured. These values are then substituted into an algebraic equation which calculates the sample's thermal conductivity. Specific directions and a reference material kit for thermal conductivity are available from TA Instruments.

Specific MDSC® Operating Parameters for Different Transitions or Properties

Glass Transition

MDSC is a much better technique than conventional DSC for measuring glass transitions. This is attributed to two major factors:

- elimination of the volume relaxation peak,

  and

- elimination of baseline slope and curvature.

To get the best results from MDSC, the following experimental conditions are recommended:

- *Amplitude = ±1°C*
  Use larger values (up to ±3°C) for very weak transitions.

- *Period = 60 seconds*

- *Heating Rate*
  Use a rate up to 5°C/min. Use lower heating rates if necessary to achieve at least four modulation cycles over the temperature range of the transition.

- Crimped aluminum pans (matched ±0.1 mg).

- Helium purge at 25 mL/min.

- 10-30 mg sample weight.
- 1 sec/point data collection rate.

Polymer Melting (Initial Crystallinity)

MDSC® has the ability to separate melting (reversing) transitions from simultaneous crystallization (nonreversing) transitions. This provides for more accurate onset temperatures, as well as more accurate and precise heats of fusion and heats of crystallization.

- *Heating Rate = 5°C/min*
- Period = 40-60 seconds
  Use longest period possible to achieve at least 4 modulation cycles at half-height of the melting peak.
- *Amplitude*
  Select maximum amplitude for "heating only" conditions as shown in Table C.4 on the next page.
- Crimped aluminum pans (matched ±0.1 mg).
- 10-15 mg sample weight.
- Helium purge at 25 mL/min.
- 1 sec/point data collection rate.

Table C.4 Maximum "Heat Only" Amplitude

Heating Rate (°C/min)

Period (sec)	0.1	0.2	0.5	1	2	5	10
10	0.003	0.005	0.013	0.027	0.053	0.133	0.265
20	0.005	0.011	0.027	0.053	0.106	0.265	0.531
30	0.008	0.016	0.040	0.080	0.159	0.398	0.796
40	0.011	0.021	0.053	0.106	0.212	0.531	1.062
50	0.013	0.027	0.066	0.133	0.265	0.663	1.327
60	0.016	0.032	0.080	0.159	0.318	0.796	1.592
70	0.019	0.037	0.093	0.186	0.372	0.929	1.858
80	0.021	0.042	0.106	0.212	0.425	1.062	2.123
90	0.024	0.048	0.119	0.239	0.478	1.194	2.389
100	0.027	0.052	0.133	0.265	0.531	1.327	2.654

$$T_{amp} = Hr * (P/2\pi * 60)$$

where:

Tamp	=	Maximum temperature amplitude for "heat only" (°C)
Hr	=	Average heating rate (°C/min)
P	=	period (seconds)
60	=	converts seconds to minutes.

Nonreversing Transitions

These include cold crystallization, enthalpic relaxations, thermoset cure, protein denaturation, and decomposition.

Use same conditions as those for melting except use an amplitude of ±1°C since cyclic heating and cooling does not affect results.

Heat Capacity & Thermal Conductivity

Modulated DSC® is unique in its ability to instantaneously measure heat capacity. This ability can be utilized in the direct measurement of thermal conductivity of some materials.

- *Heating Rate*
  Not important (0-5°C/min) except in the melting region where lower heating rates result in lower heat capacity values.

- *Period = 80-100 Seconds*
  Shorter periods can be used but sample thermal conductivity can affect results to a greater extent at shorter periods.

- *Amplitude = ±1 to 2°C*

- Calibration with material of similar C_p and thermal conductivity, using identical experimental conditions.

- Crimped aluminum pans (matched within ±0.1 mg).

- Sample weight 10-15 mg polymers; 15-20 mg sapphire; 20-30 mg metals.
- Helium purge at 25 mL/min.
- 1 sec/point data collection.

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