



TA INSTRUMENTS DIFFERENTIAL SCANNING  
CALORIMETER (DSC)

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*Insert Nickname Here*

# Operating Instructions

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## Introduction

The TA Instruments Q100 Differential Scanning Calorimeter (DSC) is a powerful and versatile thermal analyzer that allows for property measurements on a broad variety of materials from -150 to 600°C. The DSC 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, glass transitions, crystallization, oxidation, and other heat related changes. This information helps the scientist or engineer identify processing and end-use performance. The DSC instrument works in conjunction with a controller and associated software to allow for data acquisition and analysis.

## Safety

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

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch these areas or allow any material that may melt or burn to come in contact with these hot surfaces
	This symbol on the rear access panel indicates that you must unplug the instrument <i>before</i> doing any maintenance or repair work; voltages exceeding 120/240 Vac are present in this system.  If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

**Hot Stuff.** The DSC is capable of heating samples to 600°C. During testing, the furnace assembly becomes noticeably hot. After testing, the furnace assembly, sample, probe, and stage may remain hot for some time. **Do not touch the furnace assembly** or sample stage area during heating or while the sample is cooling from an elevated temperature.

**Cold Stuff.** The DSC is capable of cooling samples to  $-196^{\circ}\text{C}$  through the use of liquid nitrogen. Because of its low temperature ( $-196^{\circ}\text{C}$ ), liquid nitrogen will cause tissue damage upon contact with your skin. When you work with liquid nitrogen, use the following precautions:

- Liquid nitrogen boils rapidly when exposed to room temperature. Only use liquid nitrogen in a well ventilated area to prevent displacement of oxygen in the air.
- Wear goggles or a face shield, cryogenic gloves, long pants, and closed-toed shoes.
- 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.

**Sample Decomposition.** The DSC is capable of heating samples to  $600^{\circ}\text{C}$ . Many materials may decompose during the heating, which can generate hazardous byproducts. **WARNING:** If you are using samples that may emit harmful gases, vent the gases in an appropriate manner. In general, samples should not be heated above their decomposition temperatures to prevent the release of hazardous materials or contamination of the TMA.

## Sample Preparation

**HELPFUL HINT:** If possible, clean and dry your sample prior to DSC analysis. Unless you want to include experimental data from your fingerprints or other surface contaminants, wear gloves or use forceps when handling your DSC sample.

DSC samples should be small enough to fit in one of the aluminum DSC pans. Powder samples or flat solid samples (less than 2 mm tall) work best, although other sample geometries are allowed. Sample weight should be between **0.5 and 100 mg**.

DSC sample preparation procedures are as follows:

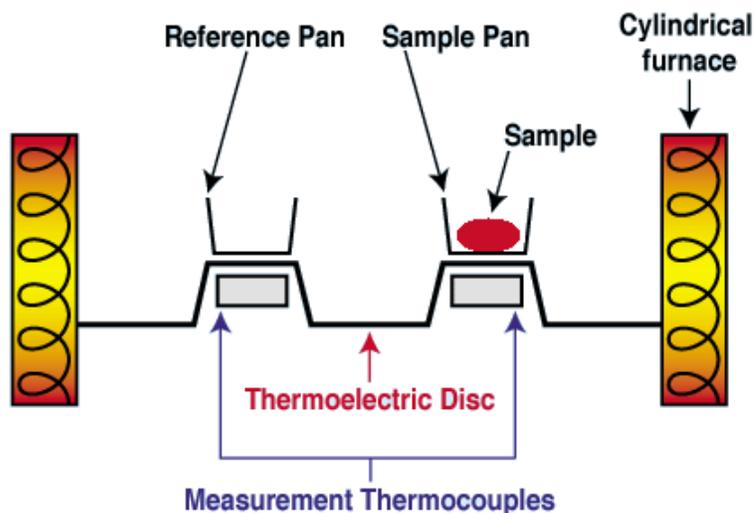
1. Weigh your sample to the nearest 0.1 mg with the analytical balance in Room 409. Record the weight.
2. Use forceps to place sample in the bottom half of an aluminum DSC pan.
3. Use forceps to place the aluminum pan lid on top of your sample.
4. Use forceps to load the aluminum pan and sample into the sample encapsulating press (Pope Jon...shown below). Align the sample pan in the encapsulating press, and press down on the handle to seal the aluminum pan.
5. Crimp an empty aluminum pan and lid. You will use the empty pan as a reference sample.



## Background

Differential scanning calorimetry (DSC) measures the energy absorbed (endotherm) or produced (exotherm) as a function of time or temperature. It is used to characterize melting, crystallization, resin curing, loss of solvents, and other processes involving an energy change. Differential scanning calorimetry may also be applied to processes involving a change in heat capacity, such as the glass transition. Similar to thermomechanical analysis, one test can generally be used to measure most or all of these properties.

In DSC analysis, the sample is placed in an aluminum pan, and the sample pan and an empty reference pan are placed on small platforms within the DSC chamber. Thermocouple sensors lie below the pans.



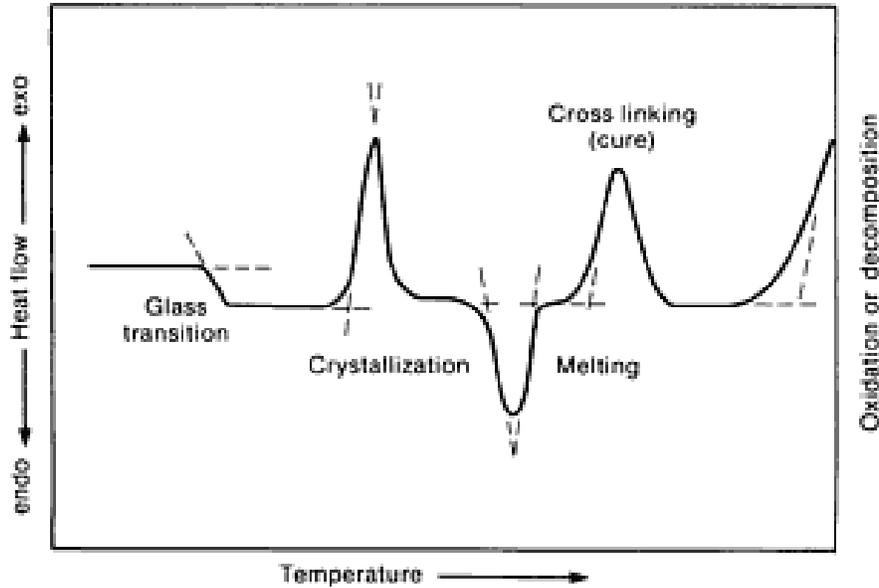
Schematic of the interior of a DSC.  
Image from <http://www.psrc.usm.edu/macrog/dsc.htm>

DSC measurements can be made in two ways: by measuring the electrical energy provided to heaters below the pans necessary to maintain the two pans at the same temperature (power compensation), or by measuring the heat flow (differential temperature) as a function of sample temperature (heat flux). The DSC ultimately outputs the differential heat flow (heat/time) between your material and the empty reference pan. Heat capacity may be determined by taking the ratio of heat flow to heating rate. Thus,

$$C_p = \frac{q}{\Delta T} \quad (1)$$

where  $C_p$  is the material's heat capacity,  $q$  is the heat flow through the material over a given time, and  $\Delta T$  is the change in temperature over that same time.

A schematic of a DSC thermogram is shown in the following figure:



Notice that this graph labels four critical points: the glass transition temperature ( $T_g$ ), the crystallization temperature ( $T_c$ ), the melting temperature ( $T_m$ ), and the curing temperature.

The **glass transition temperature ( $T_g$ )** is also an important characteristic of noncrystalline and semicrystalline materials, but  $T_g$  is a particularly significant property of many common polymers. At a temperature below  $T_g$ , amorphous and semicrystalline polymers tend to be hard and brittle because the polymer chains are locked in a tangled, coiled position. Above  $T_g$ , the polymeric chains are able to more easily rotate and slip past each other, and the polymer becomes softer and more ductile. Generally the glass transition point depends on the processing of the material, as well as that material's natural characteristics such as structure, bonding, and molecular weight. Since it takes energy to break these bonds, the glass transition appears on a DSC curve as an endothermic process. In DSC, the  $T_g$  can be found by a permanent decrease in baseline heat flow, and  $T_g$  is usually taken as the inflection point in the curve.

The **crystallization temperature ( $T_c$ )** is another important transition that occurs in some polymeric materials. At the crystallization temperature, the polymer loses its random chain arrangement, intermolecular bonds form, and the polymer molecules become more ordered. Formation of bonds during crystallization is an exothermic process, so an increase in heat flow (a peak on the DSC curve) accompanies the crystallization process. Generally, the  $T_c$  is found by finding the onset point of the crystallization curve. Note that many amorphous polymers never undergo crystallization.

**Melting** occurs when your material changes phase from a solid to a liquid. When a material starts to melt, its intermolecular bonds absorb energy and begin to loosen and break. Since melting involves absorption of energy, it is an endothermic process and appears on the DSC curve as a large, temporary decrease in heat flow. Once the material has completely melted, the heat flow returns to its original baseline value. The melting temperature is generally measured at the onset point of the melting curve.

**Curing** of a polymer occurs when individual chains form strong bonds to other chains, a process that is sometimes referred to as “crosslinking.” Like crystallization, this process of chain ordering and bond formation is exothermic. The curing temperature is generally given as the onset of the curing peak on the DSC curve.

Interpretation and analysis of DSC data may be difficult for some samples. For example, polymers containing moisture, fillers, or additives such as plasticizers may behave quite differently than “pure” polymers. In addition, some polymer properties determined by DSC may depend on the processing methods used to create or shape the materials. The TA Instruments Universal Analysis software is helpful for most baseline analyses, but you may need to do some additional interpretation of your DSC data based on your knowledge of the material and processing history.

## References

- ASM Engineered Materials Handbook Desk Edition (Online), Thermal Analysis and Properties of Polymers, Differential Scanning Calorimetry
- Askeland: Chapter 21
- <http://www.psrc.usm.edu/macrog/dsc.htm>

## 3

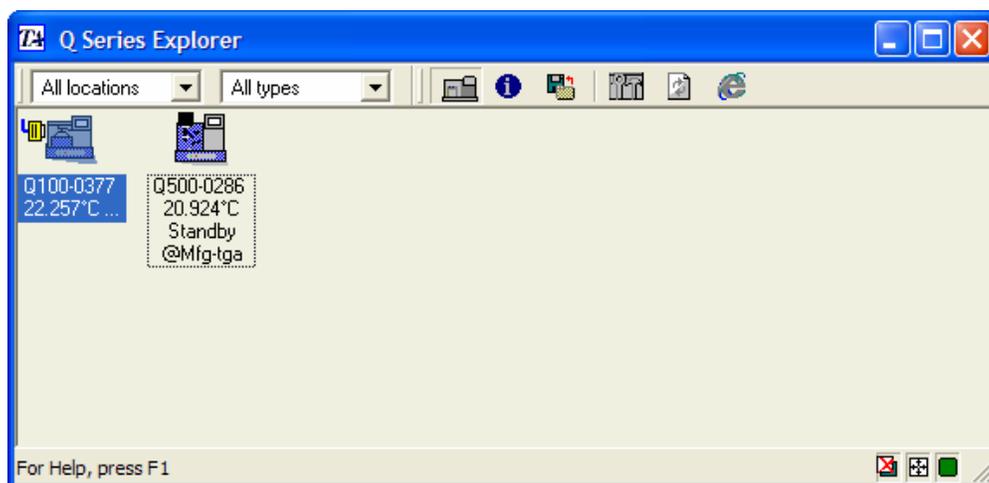
## Instrument Operation

This section will walk you through the basic operation of the DSC. You'll have to follow a series of tasks that includes

- Powering up the instrument,
- Loading your sample,
- Setting your testing conditions,
- Running a scan and collecting data, and
- Analyzing your results.

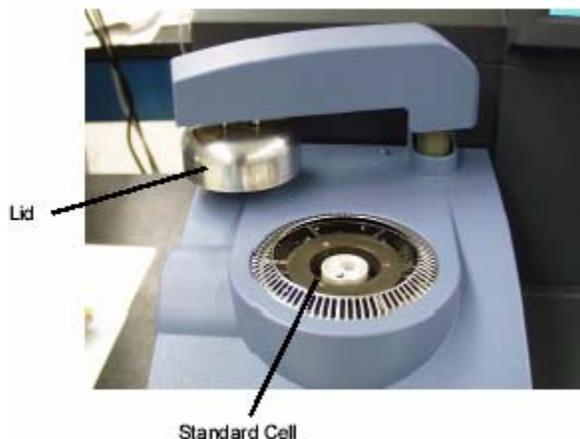
### Startup

1. Turn on the main power switch on the back of the instrument. Wait for the instrument to boot.
2. Computer: Turn on the computer. Log in using the following username and password:
  - User name: **itcltadm**
  - Password: **thermals**
3. Purge gas: Open the nitrogen cylinder main valve. If necessary, adjust the flowmeter (on the computer workbench, next to the telephone) to attain a purge gas flow rate of approximately 50 ml/min.
4. Open the control software by
  - Double clicking on the **DSC and TGA – Q Series Explorer** icon on the desktop, and
  - Double clicking on the **Q100** icon for the DSC control window.

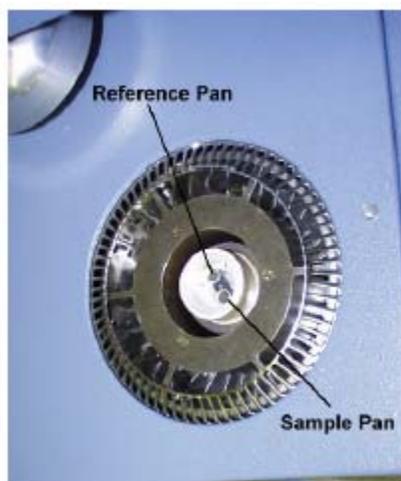


## Sample Loading

The following figure shows the DSC lid and sample cell.



This is what you'll see when you open the DSC lid. Note the two small sample pans in the center of the figure.



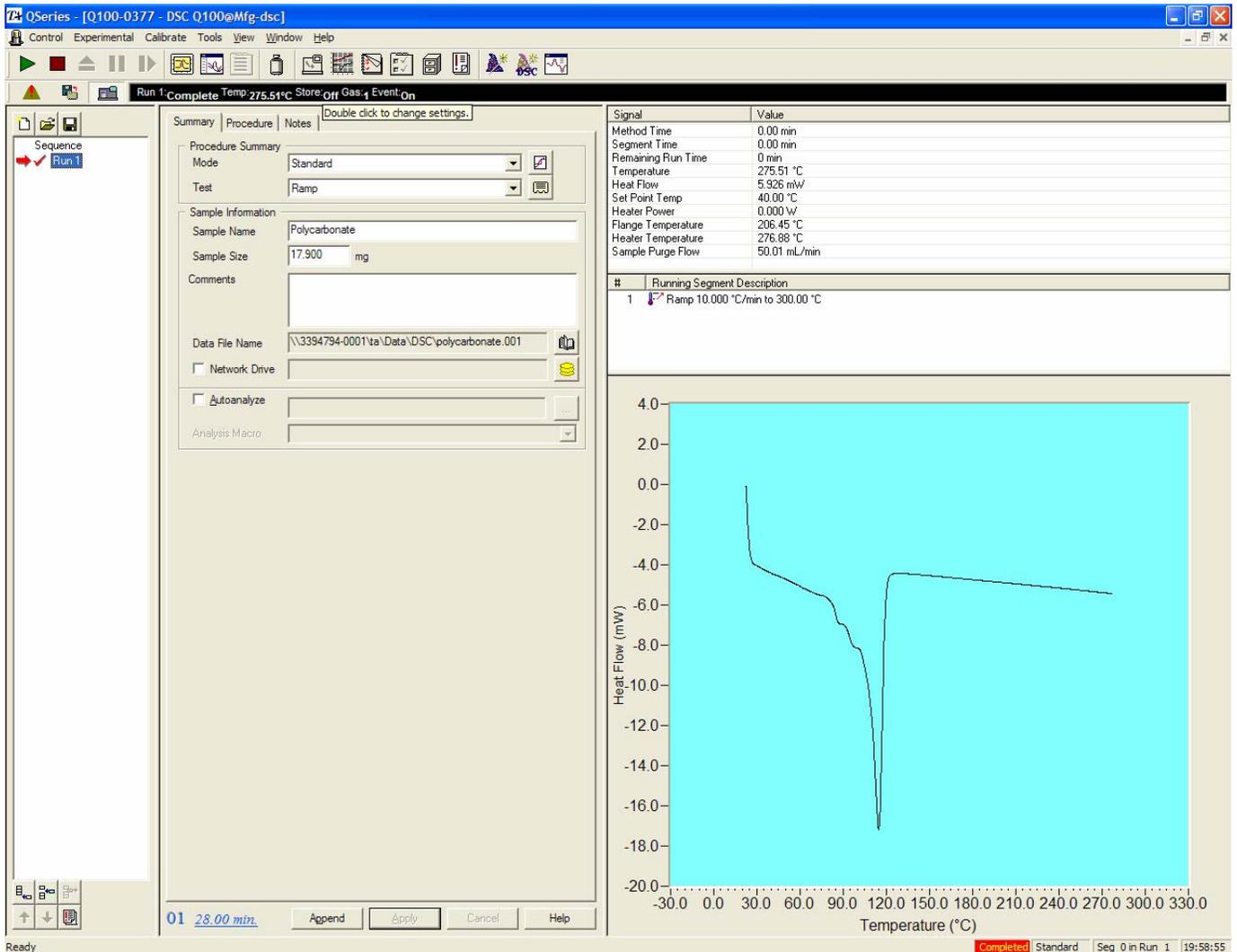
Load your sample, as follows:

1. Open the DSC by pressing the lid button on the touch screen. You will notice two small pedestals (about 8 mm diameter) on a dark bar in the center of the cylindrical sample area. If there are any aluminum pans left on the pedestals from a previous test, use forceps to carefully remove them.
2. Use forceps to **carefully** place the sample pan and empty reference pan on the two pedestals. The empty reference pan should be placed on the rear pedestal, and the sample pan should be placed on the front pedestal.
3. Close the machine by pressing the lid button on the touch screen.

## Test Conditions

The figure below shows the TMA instrument control software window.

- At the top of the window, you will notice a toolbar that includes buttons for starting (green “play” arrow) and stopping (red square) the run.
- Just below the toolbar is a status bar that includes signal displays from the DSC..
- The left frame shows the sample runs and allows you to reset the DSC after a run is complete.
- The middle frame contains tabs that allow you to change the sample information and testing parameters.
- The top right frame shows live signals from the DSC hardware.
- The middle right frame shows the run sequence for your test.
- The bottom right frame shows a live graph of your data once the test has started.



After you have loaded your sample and reference pan and closed the lid (see Sample Loading section), follow this sequence of steps to run a test:

**Summary.** This tab provides prompts for sample name, sample size, file name, etc.

1. Enter your sample name, comments, and data file name in the appropriate spaces.
2. Enter your initial sample weight in mg.

**Procedure.** This tab allows you to customize the test parameters.

1. Click on the **Procedure** tab.
2. If you would like to make changes to the heating rate or final temperature, enter the new values by clicking on the numbers in the boxes. The following settings are recommended for most specimens:
  - Start Temperature: Use current box should be checked
  - Ramp: 10.00 °C/min
  - Final Temperature: up to 600 °C (depends on the sample)
3. Click the **Apply** button to apply your changes to the current run sequence.

## Running the Test

To start the test run, simply click the green “play” arrow on the toolbar. Within moments, you should see data appearing on the graph.

## Cleanup

After the furnace and stage assemblies have cooled sufficiently, remove your sample. If you are running multiple DSC scans, you may use the same reference pan. If you are done with your testing, remove the reference pan.

**CAUTION:** The sample stage area is quite fragile and easily damaged. If you have questions regarding loading your sample or cleaning of the sample stage, ask your instructor or TA for assistance.

## Hardware Shutdown

**Turn off the nitrogen purge gas** by closing the main cylinder valve.

You may leave the instrument and computer turned on. If you would like to shut it down,

1. Shutdown the computer.
2. Turn off the main power switch on the instrument control panel.