

**Reservations:** Reserve with FBS, login to FBS to access instrument.  
Record instrument time on Log sheet.

**Safety:**

**Hot Surfaces.** The DMA is capable of heating samples to 600°C. During testing, the furnace assembly becomes noticeably hot. After testing, the furnace assembly, sample, clamp, 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 Surfaces.** The DSC is capable of cooling samples to -150°C through the use of liquid nitrogen. Because of its low temperature, liquid nitrogen will cause tissue damage upon contact with your skin.

**Getting started:**

1. Turn on the computer by logging into FBS
2. Turn on the air by rotating the lever labeled DMA on the opposite wall to be parallel to the line.
3. If the instrument is not on, push the round button on the right side of the instrument
4. On the computer, open TA Trios Software

**Choosing your parameters:**

- Clamp Geometry
  - Dual/Single Cantilever
  - Tension
  - 3-point bend
  - Shear Sandwich
  - Compression
  - Submersion Film/Fiber Tension
- Test Mode
  - Dynamic Mechanical Analysis (DMA) Testing (also known as oscillatory measurements)
  - Controlled Oscillation Strain/Stress Sweep Modes
  - Controlled Oscillation Frequency Sweep Modes at Constant Stress and Strain
  - Creep (or Step Stress) Testing
  - Stress Relaxation (or Step Strain) Testing
  - Controlled Force/Rate Testing
  - Isostrain/stress Mode
  - Strain Rate Mode

**Sample Size Dimensions per Clamp:**

Dual/Single Cantilever	8/4 mm (L), Up to 15 mm (W), and 5mm (T)
	20/10 mm (L), Up to 15 mm (W), and 5 mm (T)
	35/17.5 mm (L), Up to 15 mm (W), and 5 mm (T)
3-Point Bend	5,10, or 15 mm (L), Up to 15 mm (W), and 7 mm (T)
	20 mm (L), Up to 15 mm (W), and 7 mm (T)
	50 mm (L), Up to 15 mm (W), and 7 mm (T)
Tension Film/Fiber	5 to 30 mm (L), Up to 8 mm (W), and 2 mm (T)
Shear	10 mm square, Up to 4 mm (T)
Compression	15 and 40 mm diameter, Up to 10 mm (T)
Submersion Tension	Fixed at 15 mm (L), Up to 8 mm (W), and 2 mm (T)

### **Basic Experimental Steps:**

1. Choose, install, and calibrate the clamp appropriate for the sample shape and modulus range. Refer to TRIOS online help for information.
2. Position the thermocouple near the sample.
3. Select the mode of operation needed to perform the desired type of experiment.
4. Create a procedure that is appropriate to the operating mode, including force, frequency, heating rate, etc., as defined by the mode and the clamp type.
5. Mount the properly prepared sample on the DMA 850. Then press Measure to start the motor, preview the desired measurement, and confirm that conditions are acceptable before continuing with the experiment.
6. Close the furnace and start the experiment.

**These steps are explained in detail in TRIOS online help.**

### **Installing and Calibrating the Clamp:** See external manual for detailed instructions

1. Slide the dovetail of the moveable clamp into the dovetail holder of the drive shaft (middle post). Align the dovetail with the edge of the holder.
2. Insert the 1/16" hex key (included in the kit) to tighten the setscrew in the center of the moveable clamp. Do not overtighten the setscrew.
3. Carefully install the fixed clamp piece around the moveable clamp. The fixed clamp will attach to the four mounting posts. Line up the fixed clamp with the mounting posts and tighten the four hex screws using the torque wrench with 9/64" bit.
4. Make sure the appropriate clamp type and mode are selected in TRIOS.
5. Follow the instructions in TRIOS to calibrate the clamp.
6. Position the thermocouple so that it is close to, but not touching, the sample. (Follow the instructions in the external manual on how to move the thermocouple.

### **Creating the Test Procedure in TRIOS:**

To begin setting up a procedure, select Experiment tab > Express (for single step) or Unlimited (for multiple steps) on the Procedure toolbar.

New Experiment (Express View)

- Sample Heading: sample name, file path
- Clamp Heading: type of clamp, sample measurements
- Mode and Test Procedure: depends on clamp type
  - Mode: typically oscillation, strain control, stress control, or rate control
  - Test: typically frequency sweep, strain sweep, stress sweep, temperature sweep, temperature ramp, time sweep, or fatigue test

### **Sample Loading Basic Guidelines:**

The following general sample loading guidelines pertain to all geometries:

- Make sure the clamps are clean and free from damage.
- Remove any dust or dirt from the location surfaces at the tool mount and the geometry, since they would affect concentricity and parallelism of the geometry.
- Install the clamps correctly as described. They should be easy to install. Having to use undue force is a sign that something is wrong.
- Make sure the clamps align with each other properly.

- While loading the sample onto the geometry, ensure that the sample is centered, as well as is possible, between the mounting surfaces. Off-center loading may cause misalignment of the transducer/motor shafts. Additionally, misalignment may also affect the accuracy of the data. If misalignment does occur, the sample should be removed and carefully reloaded.
- When testing at temperatures below ambient temperature (or the temperature at which the sample was loaded), the clamps on some geometries may loosen as they cool, so it will be necessary to tighten the clamps at the minimum temperature for elastomers. To do so:
  - Monitor the temperature of the sample and wait until the sample has reached the temperature entered.
  - Open the furnace. The GCA will automatically stop and switch to a vent state and the furnace heaters will be turned off while the furnace is open.
  - Quickly and carefully tighten the clamp again.
  - Close the furnace.
  - When the temperature reaches the initial value, start the test.

#### **Removing Samples:**

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

1. Wait for the sample to return to room temperature before you attempt to remove it.
2. Unlock and open the furnace.
3. Press the Lock button to lock the moveable clamp in position.
4. Loosen the three clamping center screws that are holding the sample between the moveable jaws and remove the sample. If any sample residue remains stuck to the clamp, remove it by scraping it off with a razor blade or similar tool.

#### **Shutting Down:**

1. Remove your sample from the clamp and remove the clamp
  - a. Caution: The sample and clamp may still be hot if the furnace was used.
  - b. Press the Lock button to lock the clamp in place.
  - c. Loosen, but do not remove, the four hex screws holding the fixed clamp on the mounting posts.
  - d. Lift the fixed clamp off the four support posts. **Take care not to damage the thermocouple.**
  - e. Loosen the setscrew on the moveable clamp and then remove the clamp by sliding it out of the dovetail holder.
  - f. Place clamp in the appropriate storage box.
2. Exit from the software
3. Shutdown the instrument
  - a. To ensure proper shutdown of the instrument, press the power button on the side of the instrument and wait for the shutdown sequence.
  - b. After the instrument has completed its shutdown sequence, set the power switch on the Power Control Box to the OFF (0) position.
4. Turn off the air only to the DMA
5. Log out of FBS and record instrument time on log sheet