

Instructions for using the PerkinElmer DMA-8000

Instrument Summary:

Dynamic Mechanical Analysis (DMA) can be used to measure the relative stiffness, damping, and loss of a sample. DMA allows determination of the glass transition point, Young's modulus, shear modulus, and complex viscosity. This technique applies a periodic, sinusoidal stress or strain to a sample and measures the resulting displacement in order to determine the resulting strain or stress based response. The instrument can operate at various temperatures (*i.e.* -190-400 °C), time scales, and frequencies. Additionally, the DMA 8000 can be arranged in unique geometric configurations in order to analyze samples of varying dimensions.



Figure 1. The PerkinElmer DMA 8000.

Safety and Precautions:

- **Warning** – Check the melting point of your sample, do not surpass the melting point.
- **Warning** – When installing a sample holder, do not overtighten the fittings. The fittings should be snug without stripping the threads.
- **Warning** – Surfaces may be extremely hot or cold. Avoid touching the instrument when utilizing high temperatures or liquid nitrogen.

I. Turning on the System:

1. **Log into the Instrument** – Use the IAC's computer terminals to log into the DMA 8000.
2. **Turn on the DMA** – Flip the “**Toggle Switch**” located on the back of the instrument. Allow the instrument to initialize.
3. **Turn on Pyris Software** – Press the “**DMA**” icon on the Pyris Software banner.

II. Configuration of Instrument Geometry:

1. **Select a Geometry** – Select a geometry mode most suited for your sample. Single cantilever mode is the most common used geometry and is generally recommended if you are unsure of the proper geometry mode to use.
2. **Geometries Include:**
 - i. **Single Cantilever Mode** – Most common technique
 - ii. **Dual Cantilever Mode**
 - iii. **Three-Point Bending Mode**
 - iv. **Shear Mode**
 - v. **Tension Mode**
 - vi. **Compression Mode**
3. **Open the Instrument** – Unscrew the two finger screws located on either side of the central column, slide the locking pins outwards, and remove the chamber by lifting up.
4. **Configure the Instrument** – Use the proper fittings located in the tool box to configure the instrument. Contact the IAC's staff if you are unsure of the proper configuration.
5. **Example Configuration** – Single Cantilever mode:
 - The stationary sample fixture is placed on the geometry disk with the thermocouple located in between the sample fixture and drive shaft.
 - The sample is inserted in the clamps, about 1 mm above the thermocouple with a portion of the sample protruding outwards from either clamp.
 - **Warning** – Do not over tighten the fittings.

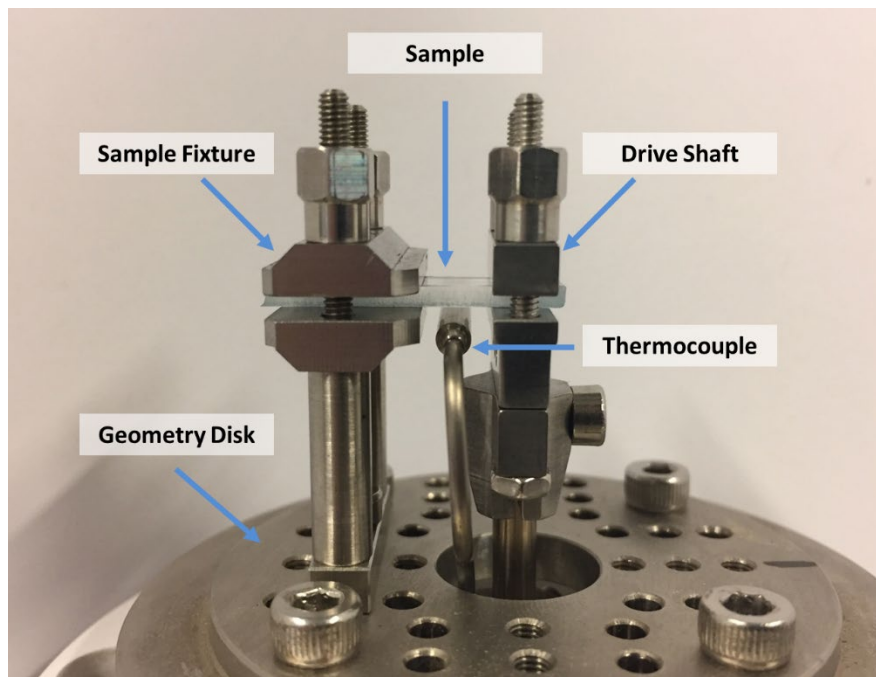




Figure 2. Example configuration of the single-cantilever geometry mode.

III. Preparing and Loading a sample:

***Note:** Before loading the sample you must zero the instrument to account for the mass of the drive shaft and geometry mode.

1. **Orient the Sample Chamber** – The instrument can rotate such that the analysis is performed at various angles. Pull the black knobs on either side of the sample column downward and rotate the column to the desired angle.
2. **Tare the Instrument** – Press the “**Balance Zero**” button:  to tare the instrument.
3. **Measure the Sample** – The instrument can generally accommodate rectangular samples that are 10-22.5 mm long. Short samples (5-10 mm) can be used; however, you will need to utilize the extended clamps.
4. **Prepare the Sample** – Cut your sample into a rectangular shape. Ensure that the edges are smooth without ridges.
5. **Mount the Sample** – Insert the sample in the instrument. Make sure the sample protrudes outwards from the clamps on either end.
6. **Replace the Sample Chamber Cover** – Place the chamber cover over the sample, slide the locking pins into place, tighten the finger screws until tight.

IV. Creating an Experimental Procedure:

1. **Create a Method** – Click the “**Method Editor**” button:  to create an experimental method. Enter the following sample info in the method editor window:
 - Sample ID
 - Operator ID
 - Comments
 - Measuring System/Geometry
 - Sample Dimensions: Length, Width, Thickness
 - File Name
2. **Enter “Initial State” Settings** – Click the initial state tab and select your pre-run settings.
 - Common Pre-Run Actions:
 - Switch gas to N₂ (20 mL/min) – Action Occurs Immediately
 - Start the Run – Action Occurs Immediately
 - Common Initial Conditions:
 - Temperature: 30 °C
 - Frequency: 0.1-10 Hz
 - Force Multiplier: 1.5
 - Static Force: 2.0 N
 - Dynamic Force: 2.0 N
 - Strain: 0.05 mm

3. Enter **“Program” Settings** – Select the program tab and enter the appropriate experimental procedure for your sample. Use the **“Add a step”** and **“Insert a step”** buttons to add experimental procedures.

- The following steps are available:
 - **Temperature Scan** – The most common used technique.
 - **Isothermal** – Analysis of fatigue behavior, curing, and degradation of a sample under constant temperature.
 - **Frequency Multiplex** – Measures material properties when the applied force is continuously changed among various frequencies.
 - **Strain Multiplex** – Measures strain dependence by measuring the force required to achieve a specified displacement.
 - **Creep/Recovery** – Measures displacement caused by applying a static load at various temperatures.
 - **Stress/Strain** – Applies a range of static stresses.
 - **Stress/Relax** – Measures the behavior of a material with constant strain.
 - **End Condition: Go to Load**
- Example temperature program conditions for a PMMA sample:
 - 1) Hold for 1 minute at 30 °C
 - 2) Heat from 30 °C to 150 °C at 2 °C per minute
 - 3) End Condition: Go to Load

4. **Check the Procedure** – Select the **“View Program”** tab and ensure that the appropriate settings are selected for your procedure. Make sure that the temperatures are appropriate for your sample.

5. **Start the Experiment** – Click the **“Start/Stop”** button to initiate the experiment.



Start/Stop Button

6. **Allow Experiment to Complete** – Allow the experiment to complete. Ensure that the temperature is at a reasonable value before handling the instrument.

V. Shut-Down Procedure:

1. **Check the Temperature** – After the experiment is complete, ensure that the temperature is near room temperature (~22 °C) before handling the instrument.
2. **Open the DMA’s Chamber** – Unscrew the finger screws on both sides of the column, slide the locking pins outward, and lift the chamber off of the column.
3. **Remove the Sample** – Remove your sample from the chamber.
4. **Reset the Sample Geometry** – Reassemble the geometry mode to single-cantilever mode.
5. **Replace the Chamber Cover** – Place the Cover over the DMA, slide the locking pins in, tighten the finger screws.
6. **Turn off the DMA** – Flip the **“Toggle Switch”** on the back of the instrument to the off position.
7. **Log-Off the Instrument** – Close Pyris. Remember to log off the instrument.

Appendix Pyris Software

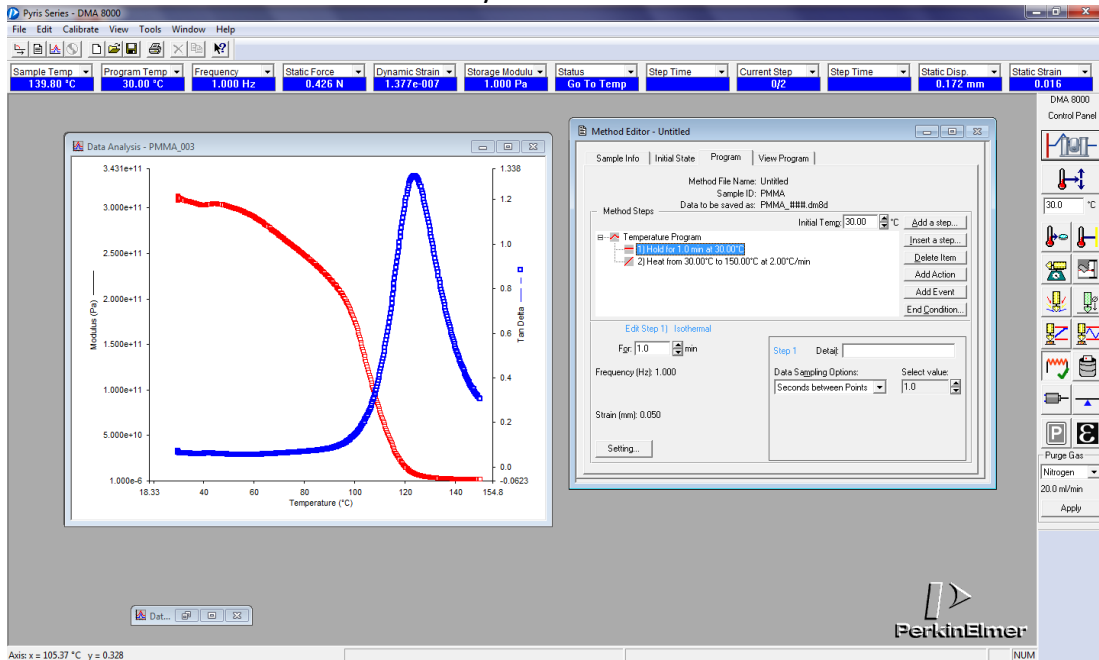


Figure 3. Pyris software window layout.

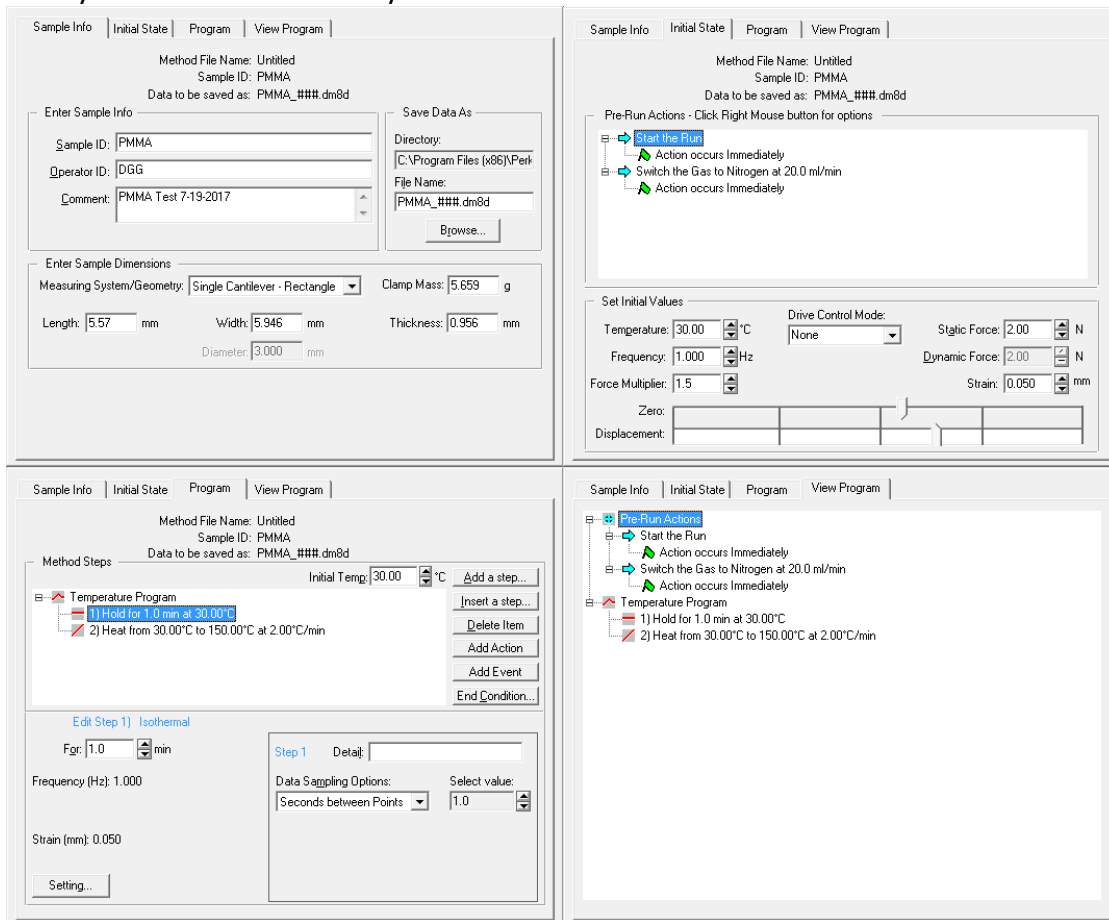
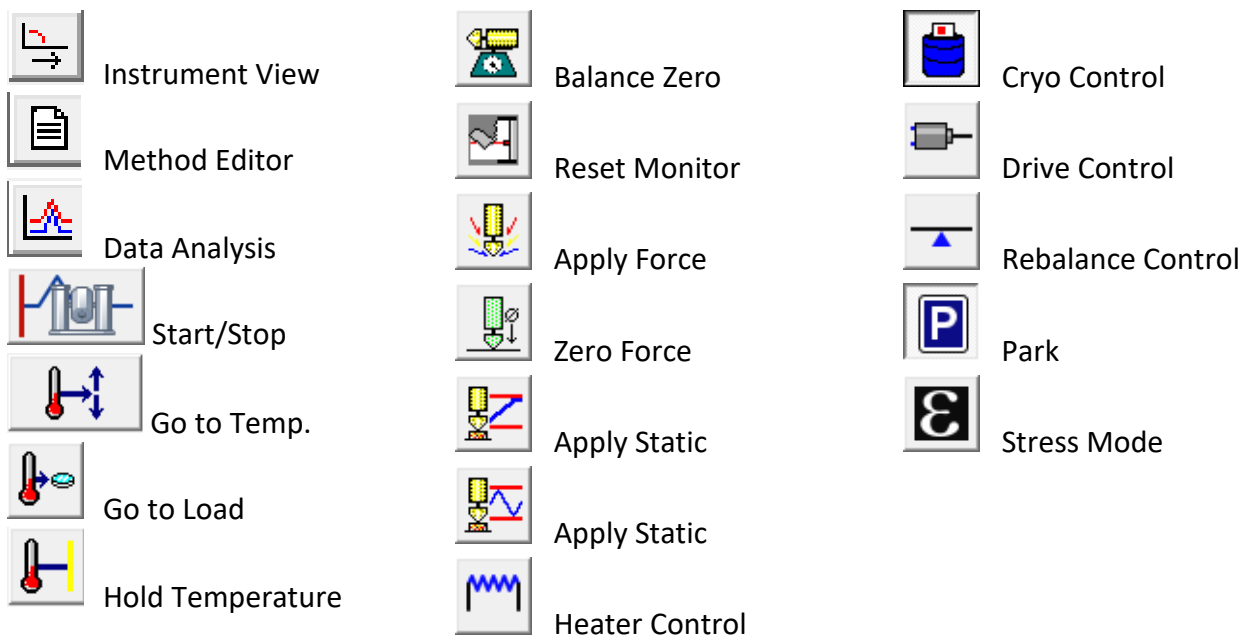


Figure 4. Method Editor window tabs.



Safety requirements

The laboratory in room ACEE 027B is a wet chemistry lab, in which some researchers may handle hazardous substances. The lab safety requires that users wear **eye protection** (safety goggles or glasses with side shields), **lab coats**, and **gloves** while using equipment in this room.

Emergency Information:

Medical Emergencies: **Contact 911 and Public Safety (609) 258-1000**

Room / facility emergencies: **Contact Public Safety (609) 258-1000**

Issues related to the instrument:

1. **Contact IAC Staff.**
2. **If unsure, leave system as is.**
3. **Try to power down the system.**

Audible/Siren Emergency Alerts:

Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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