

Operating Procedure for DMA

Purpose and Scope:

This document describes the procedures and policies for using the MSE Department Perkin-Elmer Dynamic Mechanical Analyzer. The scope of the document is to establish user procedures. Instrument maintenance and repair are outside the scope of this document. This includes the Thermolyne and the Moldatherm furnaces. This SOP does not cover the high temperature furnaces or any of the furnaces that use molydisilicide elements. Please refer to the High Temperature Furnaces SOP for those procedures.

Responsibilities:

The department Lab manager or Scientific Instructional Technician (SIT) maintains this document. The SIT is responsible for general maintenance and for arranging repair when necessary. If you feel that the instrument is in need of repair or is not operating correctly please notify the SIT immediately. The SIT will operate the instruments according to the procedures set down in this document and will provide instruction and training to users within the department. Users are responsible for using the instrument described according to these procedures. These procedures assume that the user has had at least one training session.

Definitions:

The Dynamic Mechanical Analyzer (DMA) measures the changes in mechanical behavior such as modulus and damping as a function of temperature, time, frequency, stress or a combination.

Measurements modes are: Temperature scan, time scan, modulus, tangent delta, mechanical analysis, characterization of temperature and time dependencies, frequency scan mode and creep recover mode.

Prerequisites:

All users must read this document and obtain approval and training from the SIT.

Precautions:

The DMA is a sensitive electronic device and great care should be used when handling any of the fixtures. Users should be aware that the chamber furnace could reach temperatures as high as 500 celcius. Heat sink temperature must not raise above 45 Celsius thus the need for the chiller.

The DMA shares resources with the TGA and the TMA. It is very important that the N2 gas line is connected to the TGA when it is in operation. If the TGA is not in operation and N2 is desired for the DMA measurement, you may move the line from the TGA to the DMA gas port, but you must leave a note on the TGA that includes your contact

information. All three instruments can be operated at the same time. If you are using the computer for anything other than setting up a run or extracting data and someone else needs to set up a run on another instrument, please surrender the computer.

Procedure:

Part 1: TA User Log

The DMA is part of the MSE cost center. The log MUST be filled out. It is recommended that this be done before beginning a measurement as not to forget. The Log is an Excel file located on the desktop. It should open automatically when the user first logs in.

Part 2: Operation:

Before you start, please observe the following:

Read through this procedure before starting.

Check that the right gas lines are connected and that there is enough pressure in the cylinders. If not contact Tuesday Kuykendall immediately.

Fill out the DMA log spreadsheet on desktop. Turn on the chiller. On the chiller is a breaker switch that must be turned on first, next switch to 'on' and press the start button. Do not begin any measurements until this is done.

Sample Preparation:

Most measurements are addressed in an ASTM standard and it is strongly recommended that the user consult the appropriate standard to gain information about specific sample prep for the DMA. For the purposes of this procedure, the 3pt bend test with a 15mm expanse will be assumed. Samples can be as thin as 1mm and as thick as 5mm. The default fixture is 15mm and the ratios can be 1:5:20 or 1:10:50 according to ASTM standard E-1640-04.

1. Load your sample (for 3 pt bend):
 - a. Lower the furnace if it isn't lowered yet and place a cover over the top.
 - b. If necessary, install the correct fixture for your measurement as trained.
 - c. The probe should sit on the expanse in the middle, if it doesn't the probe will need to be adjusted. Adjust probe as trained.
 - d. Place the prepared sample across the expanse.
 - e. Lower the probe and verify that it is sitting on the top of the sample; this will be the zero height.
 - f. Remove the covering and raise the furnace.
 - g. Place the collar over the furnace mouth.

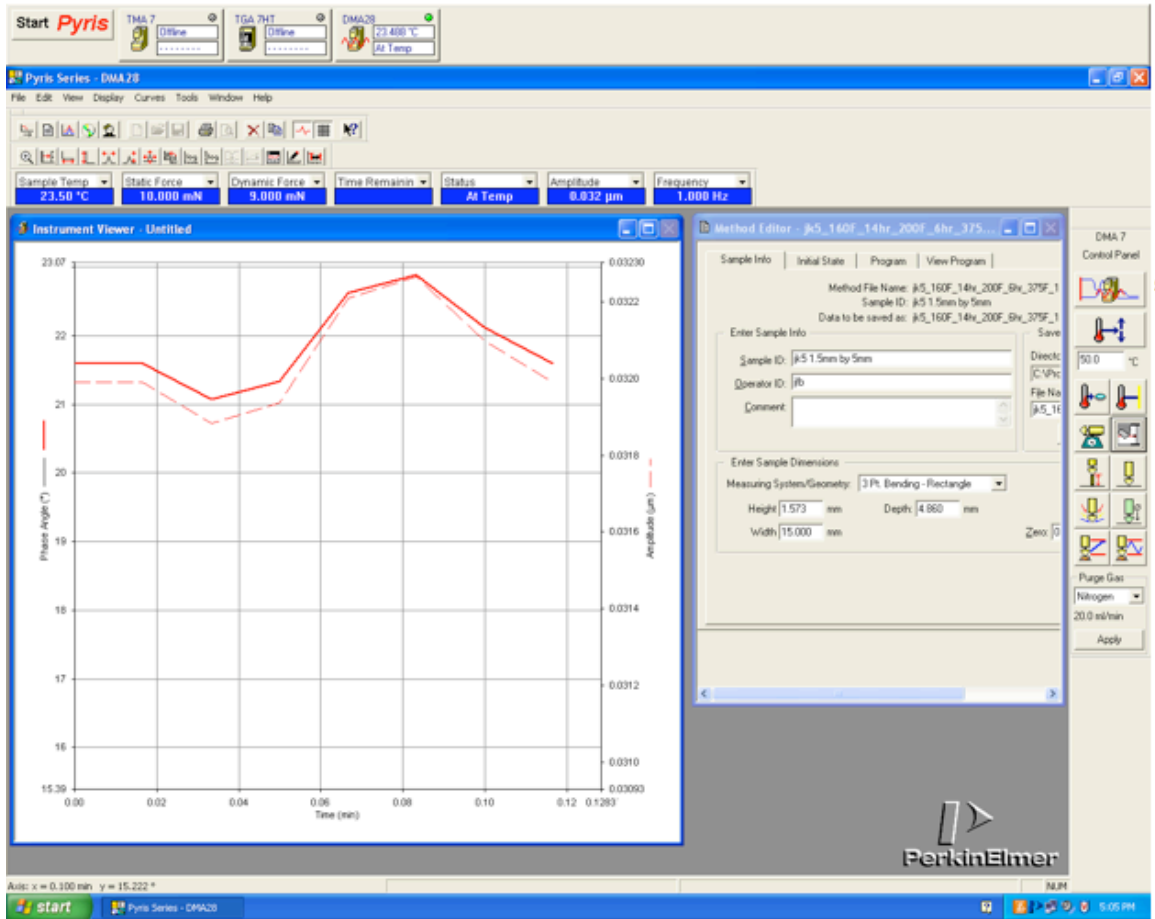
2. Open the software:
 - a. Double click on 'Pyris Manager' from the desktop
 - b. When the three applications are displayed on the top of the screen select DMA, press only once.

- c. Two dialogs will open, the “method editor” and the “instrument viewer”. At the right of the screen is a column of icons that are used to send direct commands to the DMA (see diagrams).
 - d. If one has already been prepared, open the method editor for the particular measurement.
 - e. Zero probe force
 - f. Zero probe height
 - g. Check that chiller is on
 - h. Select start
3. To create a Method:

A method can be created during the drivers test portion of the training.

 - a. Enter sample information.
 - b. Select browse to name your file and assign file path.
 - c. Select the program tab and program your scan.
 - d. What value you are measuring will determine which scan to choose for example:
 - i. Temperature Scan
 - ii. Frequency Scan
 - iii. Stress/Strain Scan
4. Note the time the scan will take to be sure the sample can be removed for other users.
5. The second dialog on the screen is the Instrument viewer. This is a live update of the force, frequency, amplitude, temperature, and time. When the scan is started, the Instrument viewer will continue to update.
6. When the scan is completed, a third dialog will be displayed, this is the data analysis graph.
7. To save the data as a .txt file, select the data analysis window, select file, select export data, and select ASCII. A dialog will open; check the box next to include data points. Your file will be saved in a folder on the desktop.

DMA Window



Sample stage and Probe

