Standard Operating Procedure: 
TA Instruments – Q800 DMA

APPROVAL REQUIREMENTS

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<tr>
<td>Author</td>
<td>RES</td>
<td>Chris Kuncho</td>
<td></td>
<td>27-FEB-15</td>
</tr>
<tr>
<td>Reviewer</td>
<td>RES</td>
<td>Patrick Casey</td>
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DOCUMENT REVISION HISTORY

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<td>A</td>
<td>Initial Release</td>
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⚠ PRIOR TO OPERATION, READ THE SAFETY PROVISIONS AND REVIEW THIS STANDARD
OPERATING PROCEDURE IN ITS ENTIRETY.

⚠ This document should only serve as a reference. The TA Instruments Q800 DMA should not be
operated by anyone without express permission and proper training by approved personnel.
SAFETY
The subsections below outline some of the obvious / major hazards that could exist during the operation of the TA Instruments Q800 DMA, and are divided to bring a level of clarity to such hazards.

**Electrical Hazards:** Electrical equipment should be installed, operated, serviced, and maintained only by qualified personnel only. Do not block the main switch as potential risk of personal injury or equipment damage if the instrument cannot be turned off in an emergency.

**Inhalation Hazards:** All materials being used should have Safety Data Sheets (SDS). Confirm that the material does not pose an inhalation hazard for the extrusion parameters being used.

**Burn Hazards:** The Furnace will be very hot. Be careful of hot surfaces with exposed skin and clothing.

**Cryogenic Hazards:** The DMA utilizes liquid nitrogen during testing. Be careful of cold/frosted surfaces with exposed skin and clothing.

**Mechanical Hazards:** Keep hands and long hair away from moving parts and feed sections. All long hair should be tied back.

**Personal Protective Equipment (PPE):** All personnel should utilize and implement proper PPE per OSHA requirements. Refer to OSHA requirements for proper use and implementation of PPE. The following items are suggested as a minimum to avoid injury based on the operation procedure outlined in this manual.

- Safety glasses
- Lab coat
- Long pants
- Close-toed shoes
- Proper gloves (when required)
INTRODUCTION

1.0 Purpose

1.1 The purpose of this Standard Operating Procedure is to set the foundation for some of the more common procedures for operating the TA Instruments Q800 Dynamic Mechanical Analysis (DMA). This includes, but is not limited to, clamp installation and calibration, as well as a brief walkthrough of one of the most common procedures for an analysis. This instrument possesses capabilities beyond a simple standard operating procedure, and will largely require some level of experimental design to use correctly and effectively. As a result it is recommended that this instrument be operated by experienced users, or under the supervision and/or guidance of an experienced user.

2.0 Description

2.1 Taken from TA Instruments Literature:

“Dynamic Mechanical Analysis (DMA) (Figure 1) is a technique used to measure the mechanical properties of a wide range of materials. Many materials, including polymers, behave both like an elastic solid and a viscous fluid, thus the term viscoelastic. DMA differs from other mechanical testing devices in two important ways. First, typical tensile test devices focus only on the elastic component. In many applications, the inelastic, or viscous component, is critical. It is the viscous component that determines properties such as impact resistance. Second, tensile test devices work primarily outside the linear viscoelastic range. DMA works primarily in the linear viscoelastic range and is therefore more sensitive to structure. DMA measures the viscoelastic properties using either transient or dynamic oscillatory tests. The most common test is the dynamic oscillatory test, where a sinusoidal stress (or strain) is applied to the material and a resultant sinusoidal strain (or stress) is measured. Also measured is the phase difference, δ, between the two sine waves. The phase lag will be 0° for purely elastic materials and 90° for purely viscous materials (Figure 1). However, viscoelastic materials (e.g. polymers) will exhibit an intermediate phase difference (Figure 2a). Since modulus equals stress/strain, the complex modulus, $E^*$, can be calculated. From $E^*$ and the measurement of δ, the storage modulus, $E'$, and loss modulus, $E''$, can be calculated as illustrated in Figure 2b. The storage modulus ($E'$) is the elastic component and related to the sample’s stiffness. The loss modulus ($E''$) is the viscous component and is related to the sample’s ability to dissipate mechanical energy through molecular motion. The tangent of phase difference, or $\tan \delta$, is another common parameter that provides information on the relationship between the elastic and inelastic components. Transient tests include creep and stress relaxation. In creep, a stress is applied to the sample and held constant while deformation is measured vs. time. After some time, the stress is removed and the recovery is measured. In stress relaxation, a deformation is applied to the sample and held constant, and the degradation of the stress required to maintain the deformation is measured versus time.”
The DMA is **VERY** sensitive and precise. Under no circumstances should the furnace and sample chamber ever be touched or manipulated without the proper tools. Serious damage to the instrumentation could occur.

![Figure 1 TA Instruments Q800 DMA](image)

**Figure 1** TA Instruments Q800 DMA

### 3.0 List of Related Internal Documents

3.1 UMass Lowell Chemical Hygiene Plan

3.2 RES-07-001 ETIC Operations Manual

### 4.0 List of Related External Documents

4.1 OSHA Laboratory Safety Guidance

4.2 QDMAGetStart

4.3 DMA
OPERATION

5.0 Safety

5.1 Safety glasses
5.2 Lab Coat
5.3 Proper Gloves (when required)
5.4 Long Pants
5.5 Close-toed shoes
5.6 UMass Lowell EEM CHP

6.0 Process Description

6.1 Instrument Specifications (Figure 2)

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
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<tbody>
<tr>
<td>Maximum Force</td>
<td>18 N</td>
</tr>
<tr>
<td>Minimum Force</td>
<td>0.0001 N</td>
</tr>
<tr>
<td>Force Resolution</td>
<td>0.00001 N</td>
</tr>
<tr>
<td>Strain Resolution</td>
<td>1 nanometer</td>
</tr>
<tr>
<td>Modulus Range</td>
<td>$10^3$ to $3 \times 10^{12}$ Pa</td>
</tr>
<tr>
<td>Modulus Precision</td>
<td>± 1%</td>
</tr>
<tr>
<td>Tan δ Sensitivity</td>
<td>0.0001</td>
</tr>
<tr>
<td>Tan δ Resolution</td>
<td>0.000001</td>
</tr>
<tr>
<td>Frequency Range</td>
<td>0.01 to 200 Hz</td>
</tr>
<tr>
<td>Dynamic Sample Deformation Range</td>
<td>± 0.5 to 10,000 μm</td>
</tr>
<tr>
<td>Temperature Range</td>
<td>-150 to 600°C</td>
</tr>
<tr>
<td>Heating Rate</td>
<td>0.1 to 20°C/min</td>
</tr>
<tr>
<td>Cooling Rate</td>
<td>0.1 to 10°C/min</td>
</tr>
<tr>
<td>Isothermal Stability</td>
<td>± 0.1°C</td>
</tr>
<tr>
<td>Time/Temperature Superposition</td>
<td>Yes</td>
</tr>
<tr>
<td>RH Control</td>
<td>Optional</td>
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</tbody>
</table>

Output Values

| Storage Modulus                | Complex/Dynamic Viscosity | Time    |
| Loss Modulus                   | Creep Compliance          | Stress/Strain |
| Storage/Loss Compliance        | Relaxation Modulus        | Frequency |
| Tan Delta (δ)                  | Static/Dynamic Force      | Sample Stiffness |
| Complex Modulus                | Temperature               | Displacement |
| Relative Humidity (Optional)   |                         |          |

Figure 2 Q800 DMA Specifications
6.2 Instrument Startup

6.2.1 The DMA should always be on. If the DMA is ever found off, check power connections in the back of the instrument. The DMA has a power toggle on its lower rear panel near the power connector (Figure 3).

![Figure 3 Q800 DMA Power Toggle](image)

6.3 If the PC is off, start up the PC by normal means.

6.3.1 Power on the Tower.

6.3.2 Power on the Monitor.

6.3 Raise the DMA furnace if closed, by pressing the raise/lower button on the TGA instrument cabinet (Figure 4).

![Figure 4 Furnace Raise/Lower Icon](image)

6.4 Sample Preparation (Figure 5).

<table>
<thead>
<tr>
<th>Dual/Single Cantilever</th>
<th>8/4* mm (l), Up to 15 mm (W) and 5 mm (t)</th>
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<tr>
<td></td>
<td>20/10* mm (l), Up to 15 mm (W) and 5 mm (t)</td>
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<tr>
<td></td>
<td>35/17.5* mm (l), Up to 15 mm (W) and 5 mm (t)</td>
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<table>
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<th>3-Point Bend</th>
<th>5, 10, or 15 mm (l), Up to 15 mm (W) and 7 mm (t)</th>
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<tbody>
<tr>
<td></td>
<td>20 mm (l), Up to 15 mm (W) and 7 mm (t)</td>
</tr>
<tr>
<td></td>
<td>50 mm (l), Up to 15 mm (W) and 7 mm (t)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Tension</th>
<th>5 to 30 mm (l), Up to 8 mm (W) and 2 mm (t)</th>
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<tbody>
<tr>
<td>Film/Fiber</td>
<td>5 to 30 mm (l), 5 denier (0.57 tex) to 0.8 mm diameter</td>
</tr>
<tr>
<td>Fiber</td>
<td>5 to 30 mm (l), 5 denier (0.57 tex) to 0.8 mm diameter</td>
</tr>
</tbody>
</table>

| Shear | 10 mm square, Up to 4 mm (t) |

![Figure 5 Q800 Sample Size](image)
6.5 Start the software. On attached PC, click the **TA Advantage Icon** (Figure 6).

**NOTE:** The Q800 DMA can be run using the touch screen on the instrument touch screen or by using the Advantage software on the PC.

![Figure 6 TA Advantage Icon](image)

6.6 Double click the Q800 instrument Icon (Figure 7).

![Figure 7 Q800 Instrument Icon](image)

6.7 The experiment screen will appear by default.

6.8 Install Clamp applicable to the experiment (Figure 8):
MÖDES OF DEFORMATION

Dual/Single Cantilever
In this mode, the sample is clamped at both ends and either flexed in the middle (dual cantilever) or at one end (single cantilever). Cantilever bending is a good general-purpose mode for evaluating thermoplastics and highly damped materials (e.g., elastomers). Dual cantilever mode is ideal for studying the cure of supported thermosets. A powder clamp is also available for characterizing transitions in powder materials.

3-Point Bend
In this mode, the sample is supported at both ends and force is applied in the middle. 3-point bend is considered a “pure” mode of deformation since clamping effects are eliminated. The 50 and 20 mm clamps on the Q800 utilize unique low-friction, roller bearing supports that improve accuracy.

Compression
In this mode, the sample is placed on a fixed flat surface and an oscillating plate applies force. Compression is suitable for low to moderate modulus materials (e.g., foams and elastomers). This mode can also be used to make measurements of expansion or contraction, and tack testing for adhesives.

Tension
In this mode, the sample is placed in tension between a fixed and moveable clamp. In oscillation experiments, the instruments use a variety of methods for applying a static load to prevent buckling and unnecessary creep. The clamps are suitable for both films and fibers.

Figure 8 Experiment Setups

6.9 Consult the following Installation and Calibration videos for each setup:

- Dual Cantilever: https://www.youtube.com/watch?v=n4akLTHqTmw
- Single Cantilever: https://www.youtube.com/watch?v=SMcWysOxmAk
- Three-Point Bend: https://www.youtube.com/watch?v=Ea9zN3RKIEg
- Film Tension: https://www.youtube.com/watch?v=Z3sCqEdwtZ0
- Compression: https://www.youtube.com/watch?v=wOyQBnMQUWE
6.10 Installing a clamp, loading a sample, and running an experiment. What follows is a walkthrough for a typical sample run using a dual cantilever clamp.

6.11.1 Raise the **Furnace** using the touch screen on the instrument (**Figure 4**).

6.11.2 Uninstall any previously installed clamps using the accompanying Allen keys such that the mounting fixture in the **Furnace** looks like **Figure 9**:

![Figure 9 Empty Mounting Fixture](image)

6.11.3 All clamp fixtures are made up of two or more parts. The **Dual Cantilever Assembly** looks as follows (**Figure 10**):

![Figure 10 Dual Cantilever Assemblies](image)

**Figure 10 Dual Cantilever Assemblies**

Center Clamp [LEFT], End Clamps [RIGHT]
6.11.4 Lock the **Air Bearing** on the DMA using the touch screen and pressing the **Float/Lock Icon** *(Figure 11).*

![Figure 11 Air Bearing Float/Lock Icon](image)

6.11.5 Install the **Center Clamp** of the **Dual Cantilever Assembly**. This can be accomplished by threading the dovetail sections and tightening the fixture by way of the Allen key and set screw. **WARNING: DO NOT OVER TIGHTEN THE SET SCREW.** The set screw typically takes just over a quarter turn to completely tighten. The assembly should look like *(Figure 12)* when finished.

![Figure 12 Center Clamp Installation](image)
6.11.6 To install the **End Clamp** of the **Dual Cantilever Assembly** line up the Allen screws with the posts on the DMA fixture as shown in **Figure 13**. Using an Allen key proceed to tighten the screws to “finger-tight.” The End Clamp Frame should have no play in it. **WARNING: DO NOT OVER TIGHTEN THE ALLEN SCREWS.**

![Figure 13 End Clamp Installation](image)

6.11.7 Using the TA Advantage software click **Calibrate** in the top menu and select **Clamp** (**Figure 14**).
6.11.8 This will bring up the DMA Clamp Calibration wizard. Here select **Dual Cantilever** and **All Calibrations** from the drop down menus, and select **Next** (Figure 15).

![DMA Clamp Calibration](image)

**Figure 14 Clamp Calibration**

**Figure 15 DMA Clamp Calibration**

6.11.9 Close the furnace using the touch screen on the instrument (**Figure 4**), and click **Calibrate**. Once the calibration has been completed, click **Next**.

6.11.10 Raise the furnace, if not already raised using the touch screen (**Figure 4**) and measure the thickness and width of the **Clamp Compliance Standard** with a set of calipers (**Figure 16**).
6.11.11 Set the **Air Bearing** to Float (**Figure 11**) and thread the **Clamp Compliance Standard** through the clamps and torque the three fixture clamps to 5.0 in/lbs. (**Figure 17**):

![Figure 16 Clamp Compliance Standard](image)

6.11.12 Using the **Telescoping Gauge** (**Figure 18**), measure the span (using a caliper to measure the output from the Telescoping Gauge) between the two sets of clamps (from each outer clamp to the center on both sides) (**Figure 19**) and adding these two values together will become the length.

![Figure 17 Installed Assembly](image)

![Figure 18 Telescoping Gauge](image)
6.11.13 Select **rectangular** from the drop down menu, and enter the observed **Length**, **Width**, and **Thickness** (**Figure 20**) of the standard into the calibration menu. Close the **Furnace** (**Figure 4**) and click **Calibrate**:

**Figure 19 Clamp Distances**

**Figure 20 Clamp Calibration**
6.11.14 Once calibration has been completed a report will be generated. Click **Finish**. This concludes the calibration portion of this walkthrough.

6.11.15 Select the type of test you wish to run, corresponding to the clamp that was installed and calibrated.

**NOTE:** The only available clamps are single and **Dual Cantilever**, **Three-Point Bend**, **Film Tension**, and **Compression**.

6.11.16 Follow the instructions on the wizard for the specific test, entering test parameters, and sample dimensions. The option to start the test will be at the end of the wizard.

6.11 Sample experiment: Here a walkthrough of a common experiment setup will be reviewed.

6.12.1 The object of this experiment is to find the $T_g$ of a sample of polycarbonate. This will be accomplished by running a fixed strain, single frequency analysis across a temperature range, from 60°C to around 180°C. The strain is one which will maintain Hookean behavior in the sample, the temperature range is one that we know the $T_g$ of polycarbonate falls between, and the frequency is one that is high enough to allow for an accurate reading, but low enough to not cause yielding of the material before the completion of the experiment.

6.12.2 Using the Advantage software, click **Experimental**, and select **Wizard** *(Figure 21)*.

6.12.3 Select **Dual Cantilever**, and select **Next** *(Figure 22)*.
6.12.4 Select **Multifrequency Modes**, and select **Next** *(Figure 23).*

**Figure 22 Starting Dual Cantilever Test**

**Figure 23 Select Multifrequency Modes**
6.12.5 Select Multifrequency (Controlled Strain) and click Next (Figure 24).

![Image of Multifrequency (Controlled Strain)]

Select the type of DMA Multifrequency mode you want to use.

- **Multifrequency (Controlled Strain)**
  
  Sample is deformed (oscillated) at a constant frequency over a range of one or more amplitudes (strains) and the mechanical properties measured.

- **Multifrequency (Controlled Stress)**
  
  Sample is deformed (oscillated) at a constant frequency over a range of one or more forces (stresses) and the mechanical properties measured.

**Figure 24 Select Multifrequency (Controlled Strain)**

6.12.7 Select Temperature Ramp/Frequency Sweep and click Next (Figure 25)

![Image of Temperature Ramp/Frequency Sweep]

Select the type of multifrequency controlled strain DMA experiment you want to perform.

- **Temperature Ramp / Frequency Sweep**
  
  Material is heated at a constant rate. While heating, the material is deformed (oscillated) at a constant amplitude (strain) over a single frequency or a range of frequencies and the mechanical properties measured.

- **Temperature Step / Frequency Sweep**
  
  Material is exposed to a series of increasing isothermal temperatures. At each temperature, the material is deformed at a constant amplitude (strain) over one or more frequencies and the mechanical properties measured. This type of experiment is recommended for time-temperature superposition (TTS) studies.

- **Isothermal Temperature / Frequency Sweep**
  
  Material is held isothermally at a user-specified temperature. Then it is deformed (oscillated) at a constant amplitude (strain) over one or more frequencies and the mechanical properties measured.

**Figure 25 Select Temperature Ramp/Frequency Sweep**
6.12.8 Select **Strain** (here we are using 0.1%). Enter the **Start Temperature** (60°C), **Soak Time** (5 min), **Final Temperature** (180°C), and **Ramp Rate** (3°C/min), and select **Next** (Figure 26).

![SOP for TA Instruments Q800 DMA](image)

Material is heated at a constant rate. While heating, the material is deformed (oscillated) at a constant amplitude (strain) over a single frequency or a range of frequencies and the mechanical properties measured.

![Temperature Ramp / Single Frequency](image)

**Figure 26** Enter Temperature Ramp Parameters

6.12.9 A review of the experiment and what parameters will be recorded during the experiment will be presented. Select **Next** (Figure 27).

![Review Recorded Test Parameters](image)

**Figure 27** Review Recorded Test Parameters
6.12.10 Enter the sample dimensions (These dimensions are obtained in the same way as those observed for the compliance standard, see 6.11.10-6.11.12), name the experiment, and designate a file save location along with a file name. Click Next once these have been completed (Figure 28). Enter the Operator and any notes and click Next (Figure 29).
6.12.11 Select **Finish** (**Figure 30**).

**Figure 30 Select Finish**

6.12.12 The screen will return to the default experimental view, and you can now start the run by clicking the green **Play** button in the upper left hand corner (**Figure 31**).

**Figure 31 Click Play to Start Experiment**

### 7.0 Maintenance

#### 7.1 Preventative Maintenance Instruction: please reference the External Documents below for Discovery TGA Maintenance
- QDMAGetStart – Maintaining the Instrument Page 62

#### 7.2 Vendor Information:

**TA Instruments**

159 Lukens Drive

New Castle, DE 19720
TRAINING CERTIFICATION CHECK SHEET

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<td>TRAINEE</td>
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<table>
<thead>
<tr>
<th>OPERATION NUMBER</th>
<th>OPERATION DESCRIPTION</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Can load software and add new experiment</td>
</tr>
<tr>
<td>2</td>
<td>Can calibrate DMA using Clamp Compliance Standard</td>
</tr>
<tr>
<td>3</td>
<td>Can define test parameters and start test</td>
</tr>
<tr>
<td>4</td>
<td>Understands the potential hazards associated with equipment</td>
</tr>
<tr>
<td>5</td>
<td>Knows the location of the UML Chemical Hygiene Plan (CHP)</td>
</tr>
<tr>
<td>6</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
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* TRAINER’S INITIAL CONFIRMS THAT TRAINEE IS PROFICIENT *