# Q Series

# Dynamic Mechanical Analyzer



**Getting Started Guide** 



#### **Notice**

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## Introduction

## **Important: TA Instruments Manual Supplement**

Please click the <u>TA Manual Supplement</u> link to access the following important information supplemental to this Getting Started Guide:

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## **Notes, Cautions, and Warnings**

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

**NOTE:** A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.



A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

## **Regulatory Compliance**

#### Safety Standards

#### For Canada

CAN/CSA-22.2 No. 1010.1-92 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

CAN/CSA-22.2 No. 1010.2.010-94 Particular requirements for laboratory equipment for the heating of materials + Amendments.

#### For European Economic Area

(In accordance with Council Directive 73/23/EEC of 19 February 1973 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN61010-1: 1993 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN61010-2-010: 1994 Particular requirements for laboratory equipment for the heating of materials + Amendments.

#### For United States

UL61010A-1 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

IEC 1010-2-010: 1992 Particular requirements for laboratory equipment for the heating of materials + Amendments.

## **Electromagnetic Compatibility Standards**

#### For Australia and New Zealand

AS/NZS 2064: 1997 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

#### For Canada

ICES-001 Issue 3 March 7, 1998 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

#### For the European Economic Area

(In accordance with Council Directive 89/336/EEC of 3 May 1989 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1: 1997 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements + Amendments. Emissions: Meets Class A requirements (Table 3). Immunity: Meets performance criteria B for non-continuous operation, minimum requirements (Table 1).

#### For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

## **Safety**



WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

AVERTISSEMENT: L'utilisateur de cet instrument est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

## **Instrument Symbols**

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

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the DMA 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.
	Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obtenir d'importantes informations relatives à sécurité. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité du système DMA.
	Si vous n'êtes pas formé aux procédures électriques, ne déposez pas les couver- cles de l'armoire sauf indications spécifiques contenues dans le manuel. La maintenance et la réparation des pièces internes doivent être effectuées unique- ment par TA Instruments ou tout autre personnel d'entretien qualifié.
4	This symbol on the rear access panel indicates that you must unplug the instrument before doing any maintenance or repair work; voltages exceeding 120/240 Vac are present in this system.
	Ce symbole apposé à l'arrière du panneau d'accès indique vous devez débrancher l'instrument avant d'effectuer des travaux de maintenance ou de réparation ; présence de tensions excédant 120/240 VCA dans cette armoire/ce circuit.
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.
<u>/ »»</u> \	Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DMA Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

### Electrical Safety

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 Vac are present in this system..



WARNING: High voltages are present in this instrument. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

AVERTISSEMENT: Présence de tensions élevées dans cet instrument. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié



WARNING: After transport or storage in humid conditions, this equipment could fail to meet certain aspects of the safety requirements of the safety standards indicated. Refer to the CAUTION on page 32 for the method of drying out the equipment before use.

AVERTISSEMENT: Après le transport ou l'entreposage dans des conditions humides, il est possible que cet équipement ne réponde pas à certains aspects des exigences de sécurité relatives aux normes de sécurité indiquées. Reportezvous à la MISE ENGARDE à la page 32 pour connaître la méthode de séchage de l'équipement avant de l'utiliser.

## Handling Liquid Nitrogen

The DMA uses liquid nitrogen as a source of cold gas in the Gas Cooling Accessory (GCA). Because of its low temperature [-195°C (-319°F)], liquid nitrogen will burn the skin. When you work with liquid nitrogen, use the following precautions:



WARNING: Liquid nitrogen boils rapidly when exposed to room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.

AVERTISSEMENT: L'azote liquide bout rapidement lorsqu'il est exposé à la température ambiante. Assurez-vous que les zones où l'azote liquide est utilisé sont bien aérées pour éviter le déplacement de l'oxygène dans l'air.

- 1 Wear goggles or a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.
- 2 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.
- 3 The purity of liquid nitrogen decreases when exposed to air. If the liquid in a container has been open to the atmosphere for a prolonged period, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

The warning below applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use.

## WARNING: Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent the Gas Cooling Accessory (GCA) in confined spaces.

Do not enter confined spaces where nitrogen gas may be present unless the area is well ventilated.

# **AVERTISSEMENT: Asphyxiant Potentiel**

L'azote liquide peut provoquer un étouffement rapide sans prévenir.

Entreposez-le et utilisez-le dans une zone bien aérée.

N'aérez pas le GCA dans des espaces confinés.

N'entrez pas dans des espaces confinés où l'azote gazeux peut être présent à moins que la zone soit bien aérée.

### Thermal Safety

During an experiment, the furnace, sample, and clamp can become very hot or very cold to the touch.



WARNING: Allow the clamp to return to room temperature before touching the clamp. Take the proper precautions when removing a hot sample or retorquing a sample.

AVERTISSEMENT: Laissez la bride de serrage revenir à la température ambiante avant de toucher la lampe. Prenez les mesures de précautions appropriées lors du retrait d'un échantillon chaud ou le resserrage d'un échantillon.



WARNING: Do not use your hands to manually move the furnace and do not put your hands up inside the furnace. It may be hot enough to cause burns.

AVERTISSEMENT: N'utilisez pas vos mains pour déplacer manuellement le four et ne levez pas vos mains à l'intérieur du four. Il peut être assez chaud pour provoquer des brûlures.

### Air Pressure Warning

WARNING: The compressed air required to operate the instrument, which is either a house air supply or supplied by the Air Compressor Accessory (ACA), is at high pressures. This high pressure can be dangerous to both personnel and equipment if not handled properly.



- If you are installing the DMA without the ACA, the tubing leading to the air filter regulator must have a pressure rating adequate to handle the source pressure. The pressure going to the air filter regulator must not exceed 1000 kPa gauge (150 psig).
- If you are installing the DMA with the ACA, the tubing supplied by TA Instruments with the accessory must be used to connect it to the air filter regulator. The ACA has a pressure relief valve limiting the pressure supplied by the ACA to 500 kPa gauge (70 psig) maximum.

The tubing supplied with the DMA must be used to connect the instrument to the air filter regulator. Set the pressure on the air filter regulator to 410 to 450 kPa gauge (60 to 65 psig).



AVERTISSEMENT: L'air comprimé requis pour l'utilisation de l'instrument, qui peut être fournipar la source d'approvisionnement en air du bâtiment ou par l'accessoire du compresseur à air (ACA), est à haute pression. Cette haute pression peut être dangereuse pour le personnel et l'équipement si elle n'est pas manipulée correctement.

- Si vous installez le DMA sans l'ACA, la tuyauterie menant au régulateur du filtre à air doit avoir une pression nominale adéquate pour la manipulation de la pression de la source. La pression qui va vers le régulateur du filtre à air ne doit pas dépasser une pression manométrique de 1000 kPa (150 psig).
- Si vous installez le DMA avec l'ACA, la tuyauterie fournie par TA Instruments avec l'accessoire doit être utilisée pour le raccordement au régulateur du filtre à air. L'ACA est équipé d'une soupape de détente de pression qui limite la pression manométrique fournie par l'ACA à 500 kPa (70 psig) maximum.

La tuyauterie fournie avec le DMA doit être utilisée pour raccorder l'instrument au régulateur du filtre à air. Réglez la pression manométrique sur le régulateur du filtre à air entre 410 et 450 kPa (60 à 65 psig).

### DMA Submersion Clamps Warning

Two DMA clamps are available for evaluation of material viscoelastic properties while the material is submerged in a fluid at temperatures up to 80°C. These clamps are designed primarily for isothermal evaluations of polymers in aqueous or buffer solutions.



WARNING: Do not use your hands to manually move the furnace and do not put your hands up inside the furnace. It may be hot enough to cause burns.

AVERTISSEMENT: N'utilisez pas vos mains pour déplacer manuellement le four et ne levez pas vos mains à l'intérieur du four. Il peut être assez chaud pour provoquer des brûlures.



WARNING: Proper clamp selection within the instrument control software (e.g., submersion film/fiber and submersion compression) is required to ensure that the 80°C upper temperature range cannot be exceeded.

AVERTISSEMENT: La sélection de la bride de serrage appropriée dans le logiciel de commande de l'instrument (p. ex. film/fibre de submersion et compression par submersion) est obligatoire afin de garantir le maintien de la plage de température la plus élevée à 80° C.

## Lifting the Instrument

The DMA is a fairly heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:



WARNING: Close the furnace before moving the instrument, even for a short distance. Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

AVERTISSEMENT: Fermez le four avant de déplacer l'instrument, même pour une courte distance. Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.

## Sample Decomposition

The DMA is capable of heating samples to 600°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 by placing the instrument near an exhaust.

AVERTISSEMENT: Si vous utilisez des échantillons qui émettent des gaz nocifs, ventilez les gaz en plaçant l'instrument près d'un échappement.

Samples should not be heated above their decomposition temperatures to prevent the relase of hazardous materials or contamination of the DMA.

We recommend that you measure the decomposition temperatures to determine the potential for problems by heating the sample materials in a thermogravimetric analyzer (TGA) or similar instrument, before running the sample on the DMA.

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# Chapter 1:

## Introducing the DMA

## Overview

The TA Instruments Q800 Dynamic Mechanical Analyzer (DMA) is an thermal analytical instrument used to test the mechanical properties of many different materials. It is the 3rd generation of DMA from TA Instruments that incorporates state-of-the-art technologies in hardware and software. To make measurements, the test specimen is mounted on one of several clamps, all of which have been designed using Finite Element Analysis to minimize mass and compliance. Basically, a deformation is imposed on the specimen in order to evaluate intrinsic as well as extrinsic mechanical properties of the material.

The DMA instrument works in conjunction with a controller and associated software to make up a thermal analysis system.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instruments.
- Enables you to set up experiments and enter necessary information.
- Stores experimental data.
- Runs data analysis programs.

**NOTE**: For technical reference information, theory of operation, and other information associated with the DMA not found in this manual, see the online help associated with the instrument control software.



Figure 1 Q Series DMA.

## DMA System Components

A functional DMA system has several major parts—the DMA cabinet, which contains the system electronics, the DMA assembly, a computer controller for analysis and control of the instrument, and an optional Gas Cooling Accessory (GCA). The following components make up the DMA assembly:

The mechanical section enclosure contains the air bearings, optical encoder, drive motor, and the associated electronics.

- The clamp assembly (called the "clamp") is interchangeable for making mechanical measurements in a variety of deformation modes to accommodate a wide array of sample shapes and materials. Several different types of clamps are available for the DMA, see page 26 for a list.
- The furnace assembly envelops the clamp assembly and provides temperature control. The furnace temperature is monitored by the control thermocouple.
- The CHROMEL®\*/ALUMEL®\* sample and reference thermocouples sense the temperature of the sample and heater and relays the readings to the instrument. The position of the sample and reference thermocouples can be changed to accommodate the various clamp assemblies.
- The DMA cabinet houses the electronics, valves, etc.
- The touch screen allows you to control and monitor some of the DMA functions from the instrument.

#### The DMA Instrument

The Q800 Dynamic Mechanical Analyzer is a precision instrument designed to measure viscoelastic properties, such as modulus and damping of rigid and soft solid materials. The sample is mounted in the clamp, one part of which is stationary while the other is movable and connected to the drive motor. Thus, the motor directly affects the deformation of the sample.

The drive motor is used to deliver force or stress to the moving drive block. This motor is non-contact in nature, in that the fixed motor assembly is not physically in contact with the movable drive block. The optical encoder measures the resulting displacement of the moving drive block. For smooth, noise-free and continuous delivery of force, the moving drive block is suspended by an air bearing.

The DMA instrument contains all the necessary electronics and software needed to perform experiments and store the results. There is a flash disk in the cabinet that saves parameters vital to system operations if power is interrupted. The touch screen enhances the overall ease of use and displays valuable information during setup, calibrations and experimentation. It has control features such as the ability to setup, start, stop or reject experiments.

The DMA was developed by TA Instruments with the following features:

- operates over a temperature range of -145°C to 600°C, using heating rates up to 20°C/min.
- determines changes in sample properties resulting from changes in seven experimental variables: temperature, time, frequency, stress, force, displacement, and strain.
- uses samples that can be in bulk solid, film, fiber, gel, or viscous liquid form.
- employs interchangeable clamps allowing you to measure many properties, including: modulus, damping, creep, stress relaxation, glass transitions, and softening points.

## The QDMA Touch Screen with QNX/Platinum<sup>TM</sup>

The DMA Q800 instrument has a built-in integrated display and keypad in the form of a touch screen for local operator control. The functions on the screen change depending upon the menu you are using. This section briefly describes the basic layout of these functions when your instrument has QNX and Platinum capabilities installed.

The status line along the top of the display shows the current instrument status, run selection, and temperature.

At the bottom of the screen is a set of keys that are used for the primary instrument functions. See the table below for a description of each key.

The functions in the middle of the touch screen will vary depending on the screen displayed.



Figure 2 DMA touch screen.

## **QNX/Platinum™ Primary Function Keys**

Use the following keys for the main functions of the instrument.

**Table 1: Primary Key Functions** 

Key Name	Description
Start	Begins the experiment. This is the same function as <b>Start</b> on the instrument control software.
Stop	If an experiment is running, this key ends the method normally, as though it had run to completion; i.e., the method-end conditions go into effect and the data that has been generated is saved. This is the same function as Stop on the instrument control software.
	If an experiment is not running (the instrument is in a standby or method-end state), the <b>Stop</b> key will halt any activity (air cool, all mechanical motion, etc.).

**Table 1: Primary Key Functions** 

Key Name	Description
Control	Displays a list of the control command functions. These are used to control the instrument actions such as furnace movement. Items can be selected from the icons or from the drop-down menu. Select Apply to initiate the command. See the next page for more details on this screen.
Display	Accesses the display screen, which displays the signals from the instrument such as signal display, real-time plot, instrument information, etc.
Calibrate	Displays the calibration functions available for this instrument. Functions such as the touch screen calibration can be accessed using this key.

## **QNX/Platinum™ Control Menu**

The Control Menu is accessed by touching the Control key at the bottom of the touch screen. A brief description of each control command is provided in the table below.

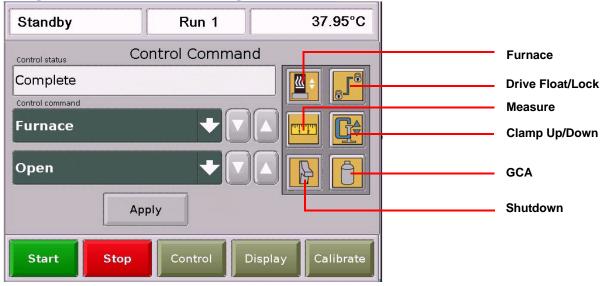


Figure 3 Control Command menu.

NOTE: Most of the commands shown are not available during an active experiment.

Select the desired function either from the drop-down list of Control Commands or by pressing the icon. Then press **Apply** to initiate the action.

**Table 2: Control Command Functions** 

Control Command	Description
Furnace	Toggles between the furnace closed (up) and furnace open (down) functions, depending on where the furnace is when you press the key. This key can be pressed while the furnace is moving, to reverse the direction of movement.

**Table 2: Control Command Functions** 

Control Command	Description
Drive Float/Lock	Toggles between floating and locking, which releases or locks the moveable clamp. <b>Floating</b> —releases the clamp, turns on the air-bearing gas, and applies zero force. <b>Locking</b> —turns off the air-bearing gas, locking the clamp in its current position.
Measure	This key toggles between active and the sample measurement. When the installed clamp is used to measure sample length (tensioning and penetration clamps), <b>Active</b> is displayed during the process of measuring the sample length or thickness (where appropriate) using the selected initial force (or a default value of 0.05 N) and turning the motor drive on. The sample measurement is displayed when the key is idle.
Clamp Up/Down	Used to raise and lower the moveable clamp by applying the initial force. If zero initial force is selected, then 0.05 N is used.  The direction of the clamp motion changes each time the key is pushed. You can stop the clamp motion by pressing the <b>STOP</b> key or the <b>DRIVE</b> key. (This function applies only to those clamps that can be raised and lowered.)
Shutdown	Shuts down and resets the instrument.
GCA Fill	Automatically fills the GCA with liquid nitrogen from the holding tank when an experiment is not in progress. This key also displays the GCA state or the level of liquid nitrogen. ("None" indicates that a GCA is not connected to the DMA.)
Air Cool	Toggles the air cool function on or off. This is the same function as Air Cool on the instrument control software.
Zero Clamp	Performs a clamp zero calibration for the tension, compression, and penetration clamps.
Rest Saved Parameters	Resets the saved instrument parameters and resets the instrument.

## Options and Accessories

Several optional clamps and accessories are available from TA Instruments to be used with the DMA. A brief description of each one follows. For more information refer to the online documentation.

## **Clamps**

The DMA utilizes several different types of clamps. These clamps can be classified as either tensioning or nontensioning clamps. Tensioning clamps require that a positive force (preload force) be placed on the sample at all times.

Tensioning clamps are:

- 3-point bending
- film tension
- fiber tension
- compression
- penetration
- submersion compression
- submersion film/fiber.

Nontensioning clamps are:

- single cantilever
- dual cantilever
- shear sandwich.

Most of the clamps have two basic parts—a moveable clamp and a fixed clamp (also sometimes called a stage).

The single/dual cantilever clamp is included with the DMA Q800. For the directions on installing and removing this clamp, refer to Chapter 2.

## **Gas Cooling Accessory (GCA)**

The GCA (see figure to the right) is used to perform subambient DMA experiments. The GCA utilizes liquid nitrogen, stored in a holding tank, to provide cooling. It has been designed for automatic refilling from a low pressure [170 kPa gauge (25 psig) maximum] bulk storage tank that can be located within 1.8 meters (6 feet) of the GCA. The GCA is also capable of being filled manually by disconnecting it from the instrument and moving it to a bulk storage source. See the GCA Getting Started Guide or online help for more information.



Figure 9 Gas Cooling Accessory.

## Air Chiller System (ACS-3) Accessory

The ACS-3 is a unique three-stage air chiller system for subambient temperature control and general cooling of the DMA Q800 with standard furnace (-100°C). The ACS-3 features low-noise durable compressors (approx. 55 dB), small footprint, uninterrupted operation, CFC-free, and for specified temperature ranges, eliminates the recurring cost and safety concerns associated with handling and use of liquid nitrogen. The ACS-3 requires an air supply at pressure of 7 bar (100 psi), flow rate of 200 l/min, and appropriate instrument-specific Air Chiller Panel. See the ACS-3 Getting Started Guide for more information.



Figure 10 ACS-3 Accessory.

## **Nitrogen Purge Cooler Accessory (NPC)**

The Nitrogen Purge Cooler (NPC), is an optional accessory for extending the temperature range of the Q800 DMA standard furnace to -160°C. The NPC consists of a 2.5 L Dewar flask that contains a copper coil tube. The furnace is cooled by purging nitrogen gas through the copper coil immersed in the liquid nitrogen-filled Dewar flask. The NPC requires a nitrogen gas supply (25 to 120 psi) at a flow rate of 30 l/min, and access to a supply of liquid nitrogen and equipment necessary for safe handling, transportation, and pouring of the liquid.

**NOTE**: The NPC is for crash cooling and controlled heating only. Controlled cooling rates are possible using the Gas Cooling Accessory (GCA), or Air Chiller System (ACS-3).



Figure 11 Nitrogen Purge Cooler Accessory.

## **Air Filter Regulator Assembly**

The air filter regulator assembly, shown in the figure to the right, is used with the instrument to help remove any oil, water, and particulates from the air being supplied to the air bearing.

The air source supplying the air filter regulator can come from a central laboratory supply or from the TA Instruments Air Compressor Accessory (ACA).

See Chapter 2 for information on installing the air filter regulator and the online help for more details on this accessory.

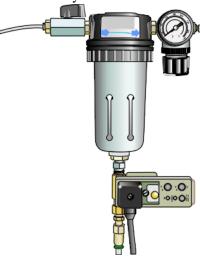


Figure 13 Air Filter Regulator Assembly.

## Instrument Specifications

The tables found on the following pages contain the technical specifications for the DMA.

**Table 8: DMA Technical Specifications** 

Item/Area	Specifications
Dimensions	Depth: 56 cm (22 in) Width: 46 cm (18 in) Height: Furnace open: 71 cm (28 in) Furnace closed: 56 cm (22 in)
Weight Weight with Transformer	39.5 kg (87 lbs) 47.7 kg (105 lbs)
Power	120 Vac, 47–63 Hz, 1.44 kVA standard 230 Vac, 47–63 Hz, 1.44 kVA if configured with a step-down transformer
Accessory Outlets	Power: 120 Vac, 47–63 Hz, 1000 VA max, combined Compatible accessories: ACA, GCA, Air Filter Regulator
Operating Environment Conditions	Temperature: 15–30 °C Relative Humidity: 5–80% (non-condensing) Installation Category II Pollution Degree 2 Maximum Altitude: 2000 m (6560 ft)
Temperature Range	−145 to 600°C
Sample Length	50 mm (2 in) maximum
Sample Width	15 mm (0.6 in) maximum
Sample Thickness	Up 5 to 10 mm (depending on clamp)
Displacement Range	25 mm (1.0 in)
Loading	0.001 to 18 N
Atmosphere	Controlled flow with inert gases or air <sup>a</sup>

a. The use of nitrogen as the air bearing gas is highly recommended when temperatures exceed  $400^{\circ}$ C.

**Table 9: Temperature Control Specifications** 

Item/Area	Specifications
Temperature Range	-145 to 600°C <sup>a</sup>
Programmed Heating Rate	0.1 to 20°C/min
Cooling Rate	0.1 to 10°C/min

**Table 9: Temperature Control Specifications** 

Item/Area	Specifications
Temperature Reproducability	±2°C
Isothermal Stability	± 0.1°C above 50°C ± 1.0°C below 50°C

a. The use of nitrogen as the air bearing gas is highly recommended when temperatures exceed  $400^{\circ}$ C.

**Table 10: Experimental Specifications** 

Item/Area	Specifications
Modulus Range	1 kPa to 3 TPa
Modulus Precision	± 1
Frequency Range	0.01 to 200 Hz
Maximum Force	18 N
Minimum Preload Force	0.001 N
Tan Delta Range	0.0001 to 10
Tan Delta Resolution	0.00001
Tan Delta Sensitivity	0.0001
Dynamic Deformation	$\pm0.5$ to 10,000 $\mu m$
Strain Resolution	1 nanometer

**Table 11: ACA Specifications** 

Item/Area	Specifications
Dimensions	Depth: 38 cm (15 in) Width: 38 cm (15 in) Height: 23 cm (9 in)
Weight (approx.)	10 kg (22 lbs)
Power Inlet Power Outlet	120 Vac 50/60 Hz 120 Vac 50/60 Hz; For use with the Air Filter Regulator only
Fuse	2 amps Slo-Blo (M)

# Chapter 2:

## Installing the DMA

## Unpacking/Repacking the DMA

The instructions needed to unpack and repack the instrument are found as separate unpacking instructions in the shipping box and in the online documentation associated with the instrument control software. You may wish to retain all of the shipping hardware, the plywood, and boxes from the instrument in the event you wish to repack and ship your instrument.



WARNING: Have an assistant help you unpack this unit. Do not attempt to do this alone.

AVERTISSEMENT: Faites-vous aider par une personne pour dépoter cet appareil. N'essayez pas de le faire tout seul.

CAUTION: Follow the directions on the unpacking instructions carefully when removing the shipping material. You MUST make sure that the four jackscrews located under the four dress cover Phillips screws are loosened properly or the instrument will not function as expected. See page 3 of the instruction sheet enclosed with your instrument, PN 985012.000, "Unpacking the Q Series DMA Instrument" for details.

MISE EN GARDE: Suivez attentivement les directives de dépotage lors de la dépose du matériel expédié. Vous devez vous assurer que les quatre vérins à vis sans fin situés sous les quatre vis à empreinte cruciforme de type Phillips sont correctement desserrés sinon l'instrument ne fonctionnera pas comme prévu. Voir la page 3 de la feuille d'instructions jointe à l'instrument,  $N^{\circ}$  de pièce 985012.000, « Dépotage de l'instrument DMA Séries Q » pour obtenir des détails.

## Installing the Instrument

Before shipment, the DMA is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual; consult the online documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts.
- Choosing a location for instrument installation.
- Connecting the DMA to the TA Instruments controller.
- Connecting cables and gas lines.
- Installing a transformer, if needed, for 230 Vac power.

It is recommended that you have your DSC installed by a TA Instruments Service Representative; call for an installation appointment when you receive your instrument.

CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

MISE EN GARDE: Pour éviter de commettre des erreurs, lisez tout le chapitre avant de commencer l'installation.

## **Inspecting the System**

When you receive your DMA, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

## **Choosing a Location**

Because of the sensitivity of DMA experiments, it is important to choose a location for the instrument using the following guidelines. The Discovery DSC system should be:

#### In

- A temperature-controlled area.
- A clean, vibration-free environment.
- An area with ample working and ventilation space.

#### On

• A stable, non-flammable work surface.

**NOTE**: Placing the DMA on a stable, vibration-free work surface is very important to instrument performance.

#### Near

- A power outlet (120 Vac, 50 or 60 Hz, 15 amps, or 230 Vac, 50 or 60 Hz, 10 amps if configured with a step-down transformer).
- Your TA Instruments thermal analysis controller (computer).
- Compressed lab air and purge gas supplies with suitable regulators.

CAUTION: Your air source must be clean, dry, and oil-free to ensure proper operation of the DMA.

MISE EN GARDE: Votre source d'air doit être propre, sèche et dépourvu d'huile afin d'assurer le fonctionnement approprié du DMA.

#### Away from

- Dusty environments.
- Exposure to direct sunlight.

- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Noisy or mechanical vibrations.

CAUTION: Drying out the instrument may be needed, if it has been exposed to humid conditions. Certain ceramic materials used in this equipment may absorb moisture, causing leakage currents to exceed those specified in the applicable standards until moisture is eliminated. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following method to dry out the DMA: 1 Ramp at 10°C/min to 400°C 2 Isothermal for 30 min.

MISE EN GARDE: Le séchage de l'instrument peut s'avérer nécessaire, s'il a été exposé à l'humidité. Certains matériels en céramique utilisés dans cet équipement peuvent absorber la moisissure, faisant en sorte que les courants de fuite dépassent ceux indiqués dans les normes applicables jusqu'à l'élimination de la moisissure. Il est important de s'assurer que la mise à la terre de l'instrument est correctement connectée à la mise à la terre des installations pour une utilisation sécurisée.

Procédez de la manière suivante pour sécher le DMA:

- 1 Augmentation de 10°C/min à 400°C
- 2 Isotherme pendant 30 min.

## **Connecting Cables and Lines**

To connect the cables and gas lines, you will need access to the DMA instrument's rear panel. All directional descriptions are written on the assumption that you are facing the back of the instrument.

**NOTE**: Connect all cables before connecting the power cords to outlets. Tighten the thumbscrews on all computer cables.

CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.

MISE EN GARDE: Chaque fois que vous branchez ou débranchez les cordons d'alimentation, tenez-les par les fiches et non par les cordons.



WARNING: Protect power and communications cable paths. Do not create tripping hazards by laying the cables across access ways.

AVERTISSEMENT: Protégez les chemins de câble électriques et de câbles de télécommunication. Ne créez pas de risques de déclenchement en posant des câbles sur les voies d'accès.

## **Ports**

The DMA has nine ports that are located on the back of the instrument. The following table provides a description of function of each port. Refer to this list when connecting cables and lines.

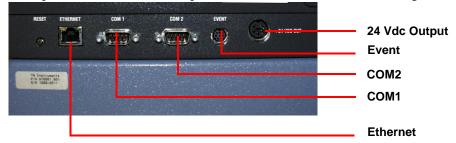


Figure 14 Ports on left rear of DMA.

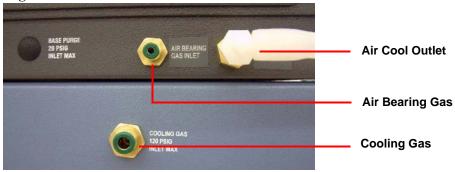


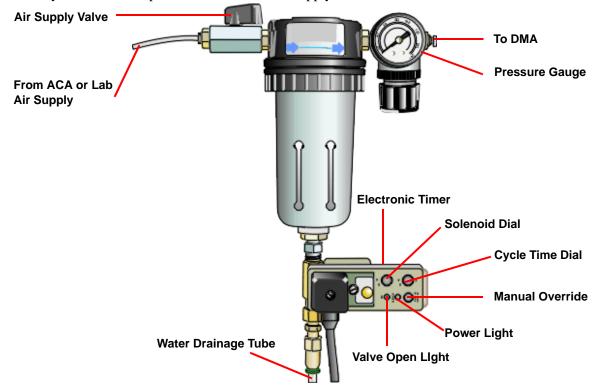
Figure 15 Ports on right rear of DMA.

**Table 12: Port Functions** 

Port	Function
Ethernet	Provides communication capabilities.
COM1	Diagnostic port (factory use only).
COM2	Accessory port. Used for GCA communications.
Event	Capable of the following functions: general purpose relay contact closure, or general purpose input 4 – 24 Vdc for external syncing. This port is not used for standard operation.
24 Vdc output	This port is not used with the DMA.
Base Purge	Not used.
Cooling Gas	Provides the furnace with air for cooling (830 kPa gauge [120 psig] maximum pressure).
Air Bearing Gas Inlet	Gas inlet port for the air bearing. Requires a clean, dry source of air or inert gas (410 to 450 kPa gauge [60 to 65 psig]).
Air Cool Outlet	Provides cool air to the furnace when the GCA is not used.

### Air Bearing Gas and Air Filter Regulator Connections

The DMA requires a 410 to 450 kPa gauge (60 to 65 psig) source of air or inert gas (e.g., nitrogen) for its air bearing system. The air source supplying the Air Filter Regulator can come from a central laboratory supply or from the TA Instruments Air Compressor Accessory (ACA). See Chapter 1 for more information about the ACA, which is an oil-less compressor. An Air Filter Regulator, shown in the figure below, is used with the instrument to supply clean, dry and oil-free gas to the air bearings. The air filter helps to remove any oil, water, and particulates from the air supply source.



**Figure 16** Air Filter Regulator.

CAUTION: Your air source must be clean, dry, and oil-free to prevent damage to the air bearings and ensure proper operation of the instrument.

MISE EN GARDE: Votre source d'air doit être propre, sèche et dépourvu d'huile afin d'assurer le fonctionnement approprié du DMA.

This gas also serves as the furnace purge. If an inert atmosphere is needed, you must use inert gas for the air bearings. The air bearings use gas at the rate of approximately 2 liters per minute. The purge gas flows through the instrument and is channeled internally to the sample.

**NOTE**: The use of nitrogen as the air bearing gas is highly recommended when temperatures exceed 400°C.

To connect the air filter regulator to the instrument, refer to the figure above and follow the instructions below.

1 Determine the air source that you will be using for the DMA. The fitting that you need on the filter depends upon which air source you intend to use as follows:

ACA air source: The air filter regulator is preassembled with a Parker quick-connect fitting in the valve on the left side of the regulator. This fitting is used with the tubing supplied with the ACA, therefore, the fitting will not need to be removed.

Lab air source: You will need to first remove the Parker quick-connect fitting in the valve on the left side of the regulator. Then install the Swagelok® fitting that is included in the accessory kit.

- 2 Place the air supply valve in the **OFF** position.
- 3 Connect the appropriate tubing, depending upon your air source, to the left side of the air filter regulator as follows:

*ACA air source*: Push the 1/8-inch tubing from the ACA (see the figure below) into the Parker fitting. Insert the tubing into the fitting until it cannot go in any further.

Lab air source: Connect 1/4-inch tubing from the air supply source to the Swagelok® fitting on the air filter regulator.

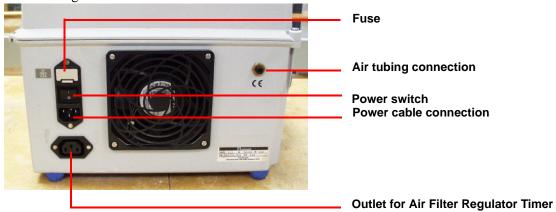


Figure 17 Air Compressor Accessory.

4 Push one end of the thin 1/8-inch tubing into the air filter regulator fitting. Insert the tubing into the fitting until it cannot go in any further. The full ten feet of tubing supplied with the ACA must be used to connect the ACA to the air filter regulator. Do not cut the tubing short, however, the tubing may be coiled to save space.

CAUTION: The full length (3 m [10 feet]) of the tubing supplied with the ACA should be used between the outlet on the ACA and the inlet to the air filter regulator. This will aid in condensing any moisture in the air and improve the effectiveness of the air filter regulator.

MISE EN GARDE: La longueur totale (3 m [10 pieds]) de la tuyauterie fournie avec l'ACA doit être utilisée entre la sortie de l'ACA et l'arrivée du régulateur du filtre à air. Cela permet de condenser toute la moisissure contenue dans l'air et d'améliorer l'efficacité du régulateur du filtre à air.



WARNING: Use of an explosive or corrosive gas as an air bearing gas is dangerous and will damage the DMA instrument. Use air or an inert gas (such as nitrogen) only, for the air bearing gas.

AVERTISSEMENT: L'utilisation d'un gaz explosif ou corrosif comme du gaz contenant de l'air est dangereuse et peut endommager l'instrument DMA. Utilisez uniquement de l'air ou un gaz inerte (tel que l'azote) pour le gaz contenant de l'air.

5 Locate the Air Bearing Gas Inlet on the right rear of the DMA instrument (see the figure below).

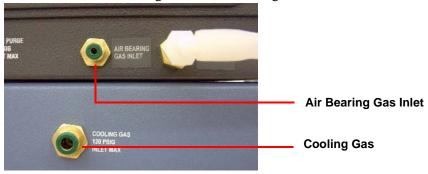


Figure 18 Air Bearing Gas Inlet.

- 6 Push the opposite end of the thin 1/8-inch tubing, which is connected to the air filter regulator, into the Legris fitting on the right side of the back of the DMA. Insert the tubing into the fitting until it cannot go in any further.
- 7 Plug the electronic timer into the Air Compressor Accessory (ACA) 120 Vac accessory power outlet, if available. If you do not have an ACA, plug the electronic timer into one of the accessory outlets on the back of the DMA. See the figure below.



Figure 19 Accessory outlets.

8 Set the dials on the electronic timer for the Auto-Drain to the desired time between drain cycles and the desired time the solenoid valve remains open during the cycle.

**NOTE**: The recommended settings are: cycle time dial set to between 20 to 30 minutes and the solenoid dial set to approximately one second.

- **9** Ensure that the filter outlet pressure is set to 410 to 450 kPa gauge (60 to 65 psig).
- 10 Turn the air supply valve on the air filter regulator to the ON position. A solenoid valve inside the DMA controls the flow to the air bearings.
- 11 If you are using the Air Compressor Accessory connected to your air filter regulator:
  - a Check the pressure gauge on the regulator before turning on the ACA. If the pressure gauge on the air filter regulator reads more than 70 kPa gauge (10 psig), release the pressure by pressing the manual override button on the electronic timer. If you try to turn on the ACA with more than 70 kPa gauge (10 psig) pressure in the system, the ACA will draw an excessive amount of current and may overload its fuse.
  - **b** Plug in the power cord on the ACA into an accessory outlet on the back of the DMA and turn it on, after you have performed step 11a.

Proper installation and maintenance of the air filter regulator is important for the performance and life of the DMA air bearings. (See the online help for information on maintaining the air filter regulator.) An efficient system ensures minimum pressure loss and removal of contaminants such as water, oil, dirt, rust, and other foreign materials. TA Instruments recommends the following minimum criteria for the air being supplied to the Air Filter Regulator:

- Oil and liquid water = < 2 mg/m3
- Water vapor dew point at 690 kPa gauge (100 psig) =  $0^{\circ}$ C (32°F)

**NOTE**: If you are using a desiccant dryer, it is best to install it after the Air Filter Regulator.

### Connecting the Cooling Gas Line

Air cooling is used to cool the DMA furnace to room temperature and heat the submersion clamp fluid only when the Gas Cooling Accessory (GCA) is not connected to the DMA. (See the GCA Getting Started Guide or the online help for further information on the GCA.)

Follow the procedure below to install the cooling gas line for air cool:

1 Locate the Cooling Gas fitting, a 1/4-inch Legris fitting on the right side of the DMA cabinet back, marked with an 830 kPa gauge (120 psig) maximum warning label.

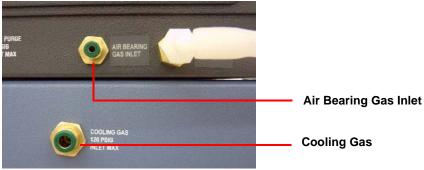


Figure 20 Cooling Gas fitting.

- 2 Make sure your compressed air source is dry, filtered, and regulated to between 170 and 830 kPa gauge (25 and 120 psig).
- 3 Connect the compressed air line to the Cooling Gas fitting.

#### Air Cool Outlet Connections

If you are using the air cool function to cool your DMA and/or heat the submersion clamp fluid, follow these instructions to make the necessary connections for air cool outlet.

Attach the Cooling Hose Accessory as follows:

Barbed fitting

Tubing connected to Air Cool Outlet and barbed fitting

Figure 21 Cooling Hose Accessory.

- a Note the shape of the cooling hose shown in the figure. Before you mount the hose, bend it to the shape shown while holding it up to the instrument to line it up with and obtain the proper distance between the fittings. The hose will retain the shape. (The purpose of this step is to eliminate any stress on the instrument frame that can be caused by bending the hose after installation.)
- **b** Use a wrench to connect the cooling hose to the fitting on the top of the bracket and to the fitting going into the instrument.
- 2 Attach a barbed fitting to the cooling hose accessory inlet as shown above.
- 3 Locate the Air Cool Outlet fitting, which is next to the Air Bearing Gas Inlet.
- 4 Connect 3/8-inch ID tubing from the Air Cool Outlet to the barbed fitting on the cooling hose accessory inlet (installed in step 2). If the Gas Cooling Accessory (GCA) is connected to the DMA, you must remove the GCA feed hose before connecting the tubing (see the GCA Getting Started Guide or the online help).

## Voltage Configuration Unit

A voltage configuration unit is required if you use 230 Vac, rather than 120 Vac. Follow these steps to install the transformer in the Power Control Unit (PCU):

WARNING: High voltages are present in this instrument as indicated by



the 1 label. Be sure to unplug the instrument before performing these instructions. See the WARNING on page 10.

AVERTISSEMENT: Présence de tension élevées dans cet instrument tel que

l'indique l'étiquette // . Assurez-vous de débrancher l'instrument avant de suivre ces instructions. Voir l'AVERTISSEMENT sur la sécurité électrique en page 10.

- 1 Remove the contents from the shipping box and verify that all of the components are present.
- 2 Remove the access plate located on the rear of the instrument by removing the four (4) screws that secure it in place.

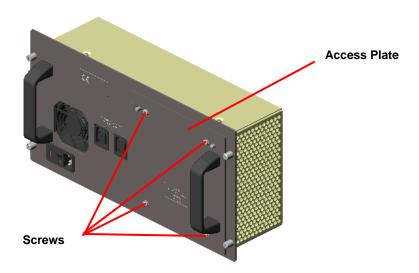


Figure 26

3 Disconnect the A10J10 connector from A10P10 located inside the PCU. Now connect the A10J10 connector on the voltage configuration unit to A10P10 located inside the PCU. Then connect A10J10

located inside the PCU to A38J1 on the anti-surge subassembly. See the diagram below for clarification.

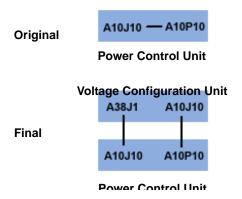
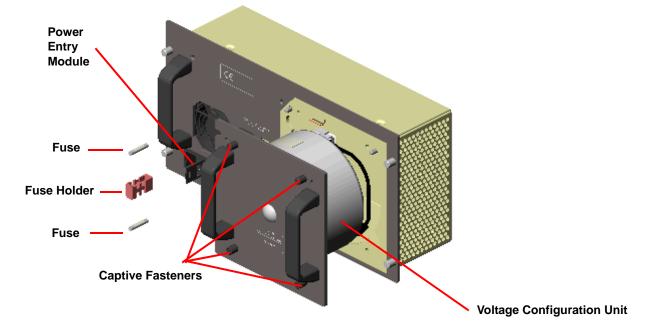


Figure 27

- 4 Install the subassembly into the PCU and tighten the four (4) captive fasteners to secure it.
- 5 Remove the fuse holder from the power entry module and replace the 10 amp fuses with 6.3 amp fuses, which are supplied in the kit. Discard the 10 amp fuses.



### Power Switch

The power switch is located at the rear of the instrument. It is part of the assembly called the power entry module, which also contains the power cable connection and fuses. The power switch is used to turn the instrument on and off.

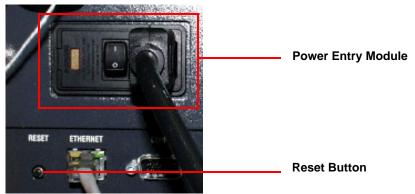


Figure 28 Power entry module.

# Power Cable

**NOTE**: A <HAR>-marked (harmonized) power cable meeting the standards of the country of installation is required for the European Economic Area.

- 1 Make sure the DMA **POWER** switch is in the Off (0) position.
- 2 Plug the power cable into the DMA power entry module.

CAUTION: Before plugging the DMA power cable into the wall outlet, make sure the instrument is compatible with the line voltage. Check the label on the back of the unit to verify the voltage.

MISE EN GARDE: Avant de brancher le câble d'alimentation du DMA dans la prise murale, assurez-vous que l'instrument est compatible avec la tension de la ligne. Consultez l'étiquette au dos de l'appareil pour vérifier la tension.

3 Plug the power cable into the wall outlet.

# Starting the DMA

- 1 Check all connections between the DMA and the controller. Make sure each component is plugged into the correct connector.
- 2 Set the instrument power switch to the ON (1) position.

After the proper power up sequence, the TA Instruments logo displays on the touch screen; this indicates that the instrument is ready for use.

**NOTE**: Allow the DMA to warm up for at least 30 minutes before performing an experiment.

# Shutting Down the DMA

Before you decide to power down your instrument, consider the following:

- All of the components of your thermal analysis system are designed to be powered on for long periods.
- The electronics of the DMA and the controller perform more reliably if power fluctuations caused by turning units on and off are minimized.

For these reasons, turning the system and its components on and off frequently is discouraged. Therefore, when you finish running an experiment on your instrument and wish to use the thermal analysis system for some other task, it is recommended that you leave the instrument on.

Should you decide to power down or reset your instrument follow these steps:

- 1 Press the **STOP** key on the touch screen to lock the drive.
- 2 Wait ten seconds to allow enough time for the system to retain the position calibration.
- 3 Shutdown the instrument as follows:
  - a To ensure proper shutdown of the instrument, it is recommended that you initiate the **Shutdown Instrument** function before turning off or resetting your instrument. This function is available on the instrument touch screen or through the Instrument Control software.
  - **b** Set the power switch to the OFF (0) position.

# Installing the Single/Dual Cantilever Clamp

When you initially receive the DMA, a clamp will need to be installed. The procedures that follow explain the installation and removal of the single/dual cantilever clamp, which is the standard clamp used on the DMA. Later, if a different sample geometry is required, you can install the appropriate clamp for the experiment.

The single/dual cantilever clamps are used to analyze weak to moderately stiff samples. The samples are rigidly clamped using the cantilever clamps.

To install the single/dual cantilever clamp on the DMA, follow these steps (refer to the figure on the next page for identification of parts):

- 1 Slide the dovetail of the moveable clamp into the dovetail holder of the drive shaft. Align the dovetail with the edge of the holder.
- Insert the 1/16 hex key on an angle as shown in the figure below to tighten the setscrew in the center of the moveable clamp. (Or use the hex key that has been shortened to allow the key to fit into the opening

on the moveable clamp.) Do not overtighten the setscrew.

- 3 Lower the fixed clamp carefully over the moveable clamp. (You may need to reposition the thermocouple. See the instructions in Chapter 3, if needed.)
- Line up the fixed clamp with the mounting posts and tighten the four hex screws.

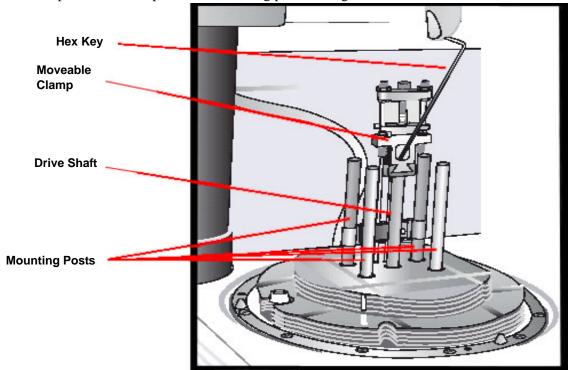


Figure 29

- 5 Ensure that the moveable clamp is aligned so that it is parallel to and equally spaced between the fixed clamps. You may need to loosen the setscrew again to adjust the moveable clamp's position. Be sure to retighten the setscrew again, if you have loosened it.
- 6 Make sure that the appropriate clamp type and mode are selected on the controller.
- 7 Calibrate the clamp for clamp mass and clamp compliance (see the online help).
- **8** Position the thermocouple so that it is close to, but not touching, the sample.

# Removing the Single/Dual Cantilever Clamp

The following procedure is used to remove the clamp.

- 1 Press the **DRIVE Float/Lock** key to lock the clamp in place.
- 2 Loosen, but do not remove, the four hex screws holding the fixed clamp on the mounting posts.
- 3 Lift the fixed clamp off the four supports.
- 4 Loosen the setscrew on the moveable clamp and then remove the clamp by sliding it out of the dovetail holder.

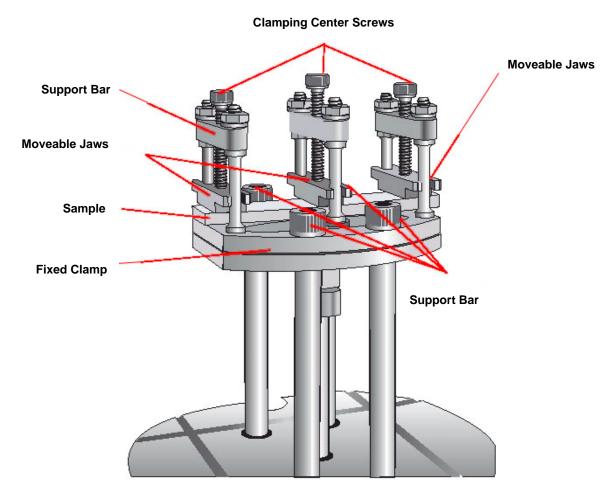


Figure 30 Dual Cantilever Clamp with sample.

# Chapter 3:

# Use, Maintenance, & Diagnostics

# Using the DMA

All of your DMA experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control software; therefore, they will not all be covered in detail here.

- Calibrating the instrument
- Selecting and preparing the sample
- Creating or choosing the test procedure and entering sample and instrument information through the instrument control software
- Loading the prepared sample and closing the furnace
- Starting the experiment

To obtain accurate results, follow these procedures carefully.

### **Before You Begin**

Before you set up an experiment, ensure that the DMA and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections from the DMA to the controller
- Connected all gas lines (air bearing and air cool)
- Connected any desired accessories
- Powered up the unit
- Become familiar with controller operations
- Calibrated the DMA, if necessary

# Calibrating the DMA

To obtain accurate experimental results, you should calibrate the DMA when you first install it. For the best results, you should recalibrate periodically. A brief description of each of the calibrations is outlines below. For details on how to perform each calibration, refer to the online documentation accessed through the instrument control software.

#### **Position Calibration**

This is used to calibrate the absolute position of the drive shaft (and slide) as read by the optical encoder. Position calibration must be done when the calibration is lost, the instrument is moved, or at least once a month.

#### **Electronics Calibration**

This procedure calibrates the instrument's electronics and drive motor over the entire frequency range of the instrument. It automatically removes the air to lock the slide (drive shaft) and applies a static force (preload force) to the motor as a calibration reference. The instrument then uses a series of frequencies to perform the calibration. Electronics calibration must be done when the DMA is moved or at least once a month.

### **Force Calibration (Balance and Weight)**

Force calibration is used to adjust the force exerted by the clamp on the sample and the force registered by the instrument as the experiment proceeds. There are two steps in the force calibration—balance (Step 1) and weight (Step 2) calibration. Force calibration must be done when the DMA is moved or at least once a month.

## **Dynamic Calibration**

Dynamic calibration is used to measure several samples of known stiffness and loss to characterize the dynamic performance of the instrument. Dynamic calibration must be done in the following situations:

- When the DMA is moved
- When the feed hose for the GCA or Air Cool is either removed or installed

or

• At least once a month.

**NOTE**: Depending on the amount of compliance samples that you use, Dynamic calibration can take from one to two hours for completion.

### **Clamp Calibration**

This procedure calibrates the properties of the installed DMA sample clamp. This calibration is performed each time you install a clamp for the first time or change from one clamp to another. Clamp calibrations may involve up to three steps (mass, zero, and compliance), depending on the clamp installed.

# Running a DMA Experiment

All of your DMA experiments will have the following general outline. In some cases, not all of these steps will be performed. See the instrument control software online help for anything not covered in this manual.

### **Basic Experimental Steps**

- 1 Choose, install, and calibrate the clamp appropriate for the sample shape and modulus range.
- **2** Position the thermocouple near the sample.
- 3 Select the mode of operation (DMA multifrequency, DMA multi-strain, DMA controlled force, etc.) 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. (Include frequency or amplitude tables when appropriate.) Pre-programmed test templates are available if you wish to use them for your experiments.
- 5 Mount the properly prepared sample on the DMA. 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 the online help.

# Choosing a Clamp Set

The shape and the modulus of most samples determine the clamp to be used for your experiment. Choosing the clamp set is obvious for some types of samples (films, for example). For others, however, there are several possible clamp sets (filled thermoplastic bars, for example). At times there can be more than one set of conditions that are necessary to make measurements on a single material over a broad temperature range. The clamping fixtures with usual material types are shown in the table below.

Once the appropriate clamp set is chosen, the DMA must be programmed to reflect the choice of clamps so that the instrument can properly control the experiment and report the correct sample moduli. The clamp type is selected from the Instrument Control software. Consult the online help for details.

This chapter provides information regarding the single/dual cantilever clamp that is the standard clamp set provided with the DMA. If you select one of the optional clamps refer to online help for details on using them.

Table 13: Clamp Sets

Clamps	Type of Sample	Examples
3-Point Bending	stiff, low damping	metals, ceramics, highly filled thermosetting polymers, highly filled crystalline, thermoplastic polymers
Cantilever (single/dual)	weak to moderately stiff	thermosetting resins, elastomers, amorphous or lightly filled thermoplastic materials

**Table 13: Clamp Sets** 

Clamps	Type of Sample	Examples
Shear	unsupported viscous liquids to elasto- mers above glass transition	uncured resins, b-staged material, tire rubber
Compression (standard and submersion)	gels and weak elastomers	personal care products, toothpaste, hydrogels
Film Tension (standard and submersion)	thin films and fibers	various types of films
Fiber Tension (standard and submersion)	single/bundled fibers	various types of fibers
Penetration	any material	various samples for DMA penetration, glass transition, or melting analysis (Not used for quantitative DMA experiments.)

# Single/Dual Cantilever Clamps

The single/dual cantilever clamps can be used for relatively weak to moderately stiff materials. The samples can range from supported thermosetting resins, to elastomers, amorphous, or lightly-filled thermoplastic materials. Dual cantilever clamps are good for testing weak elastomers and for curing supported resins. The single cantilever clamps should always be used for measuring the properties of amorphous polymers and elastomers through the glass transition, and for analyzing materials with high thermal expansion.

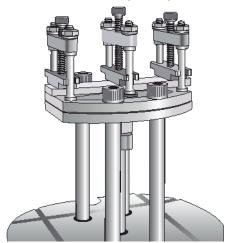


Figure 31 Single/Dual Cantilever clamps.

**Table 14: Sample Size** 

Length	Single: 4, 10, and 17.5 mm Dual: 8, 20, and 35 mm
Width	up to 15 mm
Thickness	up to 5 mm (up to 1.75 mm for best results)

**NOTE**: For the best results with the single/dual cantilever clamps, the length-to-thickness ratio should be greater than or equal to 10.

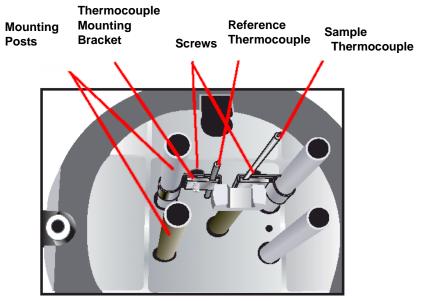
# Aligning the Thermocouples

Two thermocouples are present in the DMA furnace cavity. The right-side thermocouple provides the sample temperature reading for all clamps except the submersion compression and submersion film/fiber. Those two submersion clamps use an extended left-side thermocouple to measure the temperature of the fluid and sample.

When using any non-submersion clamp, the right-side (sample) thermocouple should be close to, but not touching the sample when it is loaded on the clamp. The left-side (reference) thermocouple need not be close to the sample and may be repositioned as necessary to clear the sample and clamp.

When using a submersion clamp, the left-side (sample) thermocouple should be bent over and the tip submerged in the fluid, close to, but not touching the bottom of the tank. Ensure that the slide can move freely without interference from the thermocouple. The right-side (reference) thermocouple need not be close to the sample and may be repositioned as necessary to clear the sample, clamp, and tank.

You may find it necessary to realign one or both of the thermocouples periodically, should they become bent or misaligned, or when a new clamp is installed. Follow these steps, referring to the figure below when needed:



**Figure 32** View from the top with clamp removed.

- 1 Loosen the screw(s) on the thermocouple mounting bracket(s)
- 2 Move the thermocouple(s) up or down, as needed, or bend to the desired angle.
- **3** Retighten the screw in the bracket(s).
- 4 Adjust the angle of the thermocouple tip, if needed, so that it is close to, but not touching the sample. You may need to bend the thermocouple in order to get it closer to the sample. Take care when you bend the thermocouple that it does not break. Do not introduce a sharp bend, a gradual bend is preferred.

# Selecting the Operating Mode

There are multiple operating modes available for the DMA. Each mode listed in the table below reflects a different class of experiment that can be performed. Before you can begin an experiment, you need to select an operating mode using the instrument control software.

#### **Table 15:**

Mode	Experiments
DMA Multifrequency Stress/ Strain	<ul> <li>time, temperature, and frequency dependence of storage and loss</li> <li>modulus, tan δ</li> <li>comparison of similar materials (modulus, transition temperature)</li> <li>thermoset curing behavior</li> <li>projection of long-term performance under load using TTS</li> </ul>
DMA Multistrain	<ul> <li>evaluation of linear viscoelastic region (LVR)</li> <li>"mullens effect" in elastomers</li> </ul>
DMA Multistress	stress fatigue     stress-dependent LVR
DMA Creep	<ul> <li>creep compliance J(t)</li> <li>material sag (elongation) under gravity (load)</li> </ul>
DMA Stress Relaxation	<ul> <li>stress relaxation modulus E(t)</li> <li>tightness of gasket seals &amp; force</li> <li>fit parts with time</li> </ul>
DMA Controlled Force	<ul> <li>stress/strain curves for films &amp; fibers</li> <li>evaluation of linear viscoelastic region</li> <li>glass transition</li> <li>softening point</li> <li>static modulus</li> <li>thermal expansion (rudimentary)</li> </ul>
DMA Isostrain	<ul><li>processing orientation effects in films</li><li>shrink force</li></ul>
DMA Strain Rate	Young's modulus     yield point

# Preparing and Mounting Samples

Sample preparation is one of the most important factors in achieving accurate and reproducible modulus values. Each type of clamp requires a specific method for sample preparation.

The procedures for the standard single/dual cantilever clamp are listed below.

### **Single/Dual Cantilever Clamp Sample Preparation**

Ideally, samples should be molded, machined, or otherwise fashioned into a rectangular shape for use with the dual cantilever clamp.

• Thickness: The minimum length-to-thickness ratio should be 10 to 1. The thickness of the rectangle should be 1/10 to 1/32 of the span of the dual cantilever clamp. The maximum thickness is 5 mm.

**NOTE**: It is very important that the sample have a uniform thickness, and that the thickness is accurately measured. The cube of the sample thickness is used in the modulus calculation; therefore, a 3% error in thickness becomes a 10% error in the calculated modulus.

- Width: The width of the rectangle should be 5 to 15 mm. The width and thickness dimensions should be uniform across the sample to within 0.02 mm.
- Length: Cut the sample 5 mm longer than the distance between the dual cantilever supports, so that the sample will lie across the supports without touching the furnace. This length is approximately 55 to 60 mm for the dual cantilever clamp and approximately 30 mm for the single cantilever clamp.

**CAUTION:** Make sure that the sample does not touch the furnace or the heating element.

MISE EN GARDE: Assurez-vous que l'échantillon ne touche pas le four ou l'élément chauffant.

Other sample shapes such as cylinders and tubes can also be used in the dual cantilever clamps, however, clamping efficiency is reduced with these shapes, and the uncertainty in modulus measurements increases as a result.

**NOTE**: The sample size must be chosen so that the sample stiffness is within the instrument's limits (102 to 107 N/m). If a sample physically fits in the clamp, it does not mean that the sample will have high or low enough stiffness for accurate measurements.

### **Operating Range for Single/Dual Cantilever Clamps**

The two equations below can help you determine which sample clamps to use and which sample sizes to choose. These equations can also help determine if the properties of a sample of a particular size can be measured or if the sample will have to be resized.

Shown on the next several pages are the modulus range versus the possible sample size range for the sample clamps. The possible sample sizes are calculated as geometry factors (GF) in the equations below. The modulus range is based on the range of stiffness over which the DMA can operate  $(10^2 \text{ to } 10^7 \text{ N/m})$ .

Single Cantilever: 
$$GF \ = \ \frac{1}{F} \left[ \frac{L^3}{12I} + 2S(1+\nu) \frac{L}{A} \right]$$

Dual Cantilever: 
$$GF = \frac{1}{F} \left[ \frac{L^3}{24I} + S(1+v) \frac{L}{2A} \right]$$

where: L = sample length of one side (mm)

A = sample cross sectional area (mm2)

I = geometric moment (mm4) = 1/12 T3W for rectangular samples

T = sample thickness (mm) W = sample width (mm)

F = clamping factor (nominally 0.9) S = shearing factor (nominally 1.5) v = Poisson's ratio (nominally 0.44).

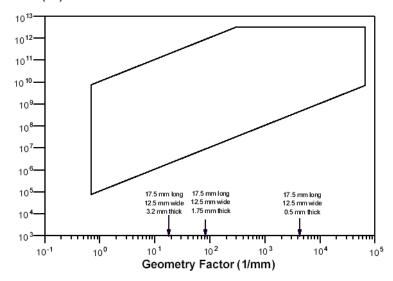
Figure 33 Geometry Factor Equations.

**NOTE**: Refer to the "Clamping Factors" section in the online help determine the value used for the clamping factor, F.

These equations are explained in greater detail in online help.

The next two figures illustrate some typical sample geometries.

#### Modulus (Pa)



**Figure 34** Operating Range of the Single Cantilever Clamps. **Modulus (Pa)** 

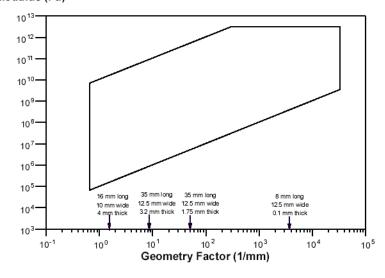


Figure 35 Operating Range of the Dual Cantilever Clamps.

### Use of Operating Range Figures

The previous figures can help to determine the proper sample shape for a given material, or to determine if a sample of a particular size can be measured. To determine the proper sample size for a specific material requires some knowledge of the approximate behavior (modulus) of the material to be tested. For example, a given material may have a modulus approximately  $3x10^9$  Pa at room temperature and will go to  $10^7$  Pa above Tg.

From the figures on the previous page, it can be seen that most geometry factors can accommodate the material at room temperature, but only a limited range of geometries ( $10^0$  to  $10^2$  1/mm) can accommodate the sample above glass transition.

The second use for those figures is to determine whether a sample of a particular size can be used. For example, you may have a sample that is 12.5 mm wide by 3.2 mm thick, with the standard (35 mm) cant lever clamps. The geometry factors for both the single cantilever (17.5 mm) and dual cantilever (35 mm) clamps are displayed on the above figures. If the material has relatively low modulus (i.e.,  $10^6$  Pa), at room temperature, then the dual cantilever clamps can easily accommodate this sample, but this sample is at the lower edge of the operating range of the single cantilever clamps. If accurate information above room temperature is desired, then the dual cantilever clamps would be preferred, or a smaller (10 mm or 4 mm) single cantilever clamp would be needed.

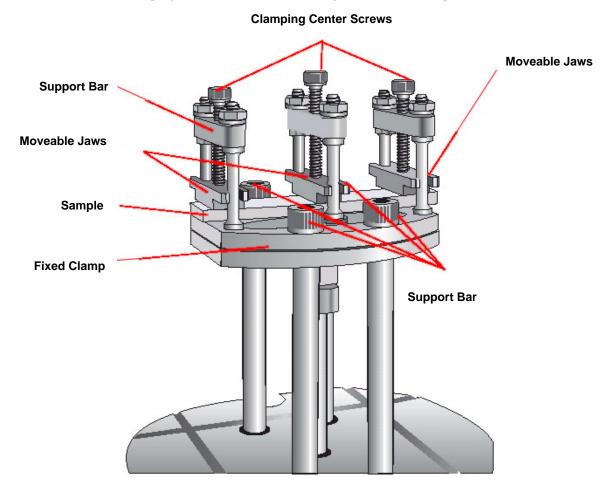
### Single/Dual Cantilever Clamp Sample Mounting

Two procedures are provided in this section—one to mount stiff samples and one to mount weak samples. Refer to the section appropriate to the type of sample you are using.

#### Mounting a Stiff Sample

To mount a stiff sample on the single or dual cantilever clamps, follow these steps:

1 Loosen the three clamping center screws (two for single cantilever clamp).



**Figure 36** Dual Cantilever Clamp with sample.

2 Press the **DRIVE Float/Lock** key to release (float) the moveable clamp.

3 Lifting each one of the moveable jaws in turn, slide the sample in from one side between the clamp faces.

**NOTE**: If the thermal expansion of a material is high, use the single cantilever clamp, rather than the dual cantilever clamp, to obtain the most accurate results. Use the rear fixed clamp for single cantilever operation to place the sample closer to the thermocouple.

- 4 Tighten the clamping screws on the fixed clamp until they are finger tight.
- 5 Tighten the clamping screw on the moveable clamp until it is finger tight.
- 6 Press the **DRIVE Float/Lock** key again to lock the clamp in position.
- 7 Use the torque wrench to tighten each of the clamping screws to maximize clamping but minimize sample deformation. Suggested clamping torques are 1.1 N-m (10 in-lb) for high modulus materials (E' > 5 GPa), 0.6 to 0.9 N-m (5 to 8 in-lb) for thermoplastic samples (E' ~ 1 GPa) and finger tight for most elastomers above Tg.
- **8** Adjust the thermocouple so that it is approximately 1 mm below and 1 mm to the side of the sample.

CAUTION: Make sure that the sample does not touch the furnace or the heating element.

MISE EN GARDE: Assurez-vous que l'échantillon ne touche pas le four ou l'élément chauffant.

#### Mounting Weak Materials, Including Resins or Other Materials, on the Fiberglass Braid

If you are using the single or dual cantilever clamps to analyze resins or some other sample on the fiberglass braid, we recommend using the 8-mm dual cantilever clamps. Follow the steps below to mount samples on the clamp:

- 1 Perform steps 1 through 6 in the previous section using a rigid sample (such as one of the steel compliance samples).
- 2 Loosen the clamping center screws and remove the rigid sample.
- 3 Lifting each one of the moveable jaws in turn, slide paperclips or other small items between the jaws to prop the clamp faces open. We recommend that aluminum foil be placed over each clamp face to prevent the resin from sticking the jaws shut.
- 4 Prepare the braid sample with the resin, spreading the resin thinly and uniformly onto the braid.
- 5 Carefully slide the prepared braid in from one side of the clamp.
- **6** Remove the paperclips or other propping devices from between the clamp faces.
- 7 Tighten the clamping center screws to finger tightness. Use a hex key to tighten the screws an additional 1/4 turn.
- **8** Adjust the thermocouple so that it is approximately 1 mm below and 1 mm to the side of the sample.

## **Measuring Sample Length**

To obtain accurate modulus values, it is important that the sample dimensions be measured accurately.

• When using the dual cantilever clamp, the sample length is defined by the length between the two fixed clamps, minus the moveable clamp's thickness. This measurement becomes fixed, i.e., 35 mm, and can be used without the need for verification.

• When using the single cantilever clamp, the sample length is defined by the length between the fixed and moveable clamps. However, due to the flexibility of the drive shaft, after the sample is tightened in the clamps, the actual sample length may be slightly different from the default values. To accurately measure the sample length, a telescoping gauge is provided in the accessory box. The next section provides the instructions needed to use the telescoping gauge.

### Using the Telescoping Gauge

To accurately measure sample length using the telescoping gauge on the single cantilever clamp, follow these steps:

- 1 Prepare and mount the sample as directed in this chapter.
- 2 Loosen the gauge tips by turning the knurled knob counterclockwise approximately one-quarter turn.



Figure 37 Telescoping Gauge.

- 3 Insert the gauge sideways between the clamps, just above the sample.
- 4 Gently rotate the gauge so that the spring-loaded gauge tips are exactly perpendicular to the clamp jaws and at the middle of the sample. Make sure that you keep the handle vertical.
- 5 Set the gauge by turning the knurled knobs clockwise to lock the tips in position.
- **6** Rotate the gauge sideways and remove it from the clamps.
- 7 Measure the gauge length, which is equal to the actual sample length, with calipers. Use this value as your sample length when entering the instrument parameters.

# Performing Experiments

Once you have set the appropriate instrument and experimental parameters and have mounted a sample on the DMA, you are ready to run the experiment.

When you run experiments using the single or dual cantilever clamps, follow the instructions given in the next section. Please note the following conditions pertaining to single/dual cantilever clamp experiments:

- If the thermal expansion of a material is high—such as for thermoplastics and rigid elastomers—use the single cantilever clamp, rather than the dual cantilever clamp, to obtain the most accurate results.
- You may need to tighten the clamps at the minimum temperature, when you run elastomer samples at subambient temperatures. To do this, follow the suggested method below:
- 1 Install and calibrate the desired clamp.
- 2 Program the desired test method, including an Equilibrate segment as your first segment.

**NOTE**: Use the Measure Again After Method Equilibration option on the Advanced Parameters window to adjust for dimension changes in the sample after the sample temperature equilibrates at the lower temperature setting. This option is applicable to film/fiber tension, compression, and penetration clamps only.

- 3 Load the desired sample.
- 4 Tighten the clamp and press **FURNACE** on the touch screen to close the furnace.
- 5 Select **Control/Go To Temperature** from the menu and enter the desired low starting temperature. Click the **Set** button. This will bring your sample to the starting temperature without applying forces to it that might distort it.

**NOTE**: The desired temperature must be within a range of –180 to 20°C.

- 6 Observe the temperature of the sample and wait until the sample has reached the temperature entered.
- 7 Press **FURNACE** on the touch screen to 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.
- **8** Quickly and carefully tighten the clamp again.

CAUTION: Use the appropriate tools and safety precautions if you need to handle the sample or clamps. They can be hot or cold enough to cause injury.

MISE EN GARDE: Utilisez les outils et les précautions de sécurité appropriés si vous devez manipuler l'échantillon ou les brides de serrage. Ils peuvent être assez chauds ou froids pour provoquer des blessures.

- **9** Press **FURNACE** on the touch screen to close the furnace.
- **10** Select START to begin your programmed experiment.

### **Starting an Experiment**

Before you start the experiment, ensure that the DMA is connected with the controller and you have entered all necessary information through the instrument control software.

**NOTE**: Once the experiment is started, operations are best performed at the computer keyboard. The DMA is very sensitive to motion and might pick up the vibration caused by touching a key on the instrument touch screen.

### **Stopping an Experiment**

If for some reason you need to discontinue the experiment, you can stop it at any point by selecting **Stop** through the instrument control software or by pressing **STOP** on the touch screen.

### **Removing Samples**

When the experiment has run to completion, remove the sample from the single/dual cantilever clamp as follows:

- 1 Wait for the sample to return to room temperature before you attempt to remove it.
- 2 Press the **FURNACE** key to raise the furnace.
- 3 Press the **DRIVE Float/Lock** key or STOP 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.

# Maintaining the Instrument

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the online documentation installed with the instrument control software for further information.



DANGER: Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

DANGER: À cause de la présence de tensions élevées dans cet instrument, le personnel non formé ne doit pas essayer de tester ou de réparer les circuits électriques.

### **Cleaning the Touch Screen**

You can clean the DMA touch screen as often as you like. The touch screen should be cleaned with a household liquid glass cleaner and soft cloth. Wet the cloth, not the touch screen, with the glass cleaner and then wipe off the screen and surrounding surfaces.



WARNING: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the user interface screen, as you may scratch the surface and degrade its properties.

AVERTISSEMENT: N'utilisez pas de produits chimiques agressifs, de nettoyants abrasifs, de la laine d'acier ou tout autre matériau rugueux pour nettoyer l'écran de l'interface utilisateur, car vous pourriez égratigner sa surface et dégrader ses propriétés.

### **Replacing Fuses**



WARNING: Always unplug the instrument before you examine or replace the fuses.

**AVERTISSEMENT**: Débranchez toujours l'instrument avant d'examiner ou de remplacer les fusibles.

The DMA contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only customer-replaceable fuses are the fuses located in the power entry module located at the rear of the instrument. To check or change these fuses:

- 1 Turn the instrument off and remove the power cord.
- 2 Insert a small screwdriver at the edge of the power entry module door and pry it open.
- 3 Insert the screwdriver on the edge of the fuse holder to pull it out of the instrument.
- 4 Remove old fuses and replace the fuses only with the type and rating indicated on the instrument's rear panel.
- 5 Place the fuse holder back into the opening and push the door shut.

# Replacement Parts

Replacement parts for the DMA that are available from TA Instruments are listed below. Refer to the tables below when ordering parts.

Table 16: DMA Accessory Kit (985011.901) Items

Part Number	Description
985011.901	DMA Accessory Kit (includes all items below)
280037.000	Tool, wrench torque drive
982161.002	Sample, ABS (Acrylonitrile Butadiene Styrene) 6 cm (2.35 inch) (5)
982161.003	Sample, ABS (Acrylonitrile Butadiene Styrene) 3.5 cm (1.4 inch) (5)
982165.002	Sample, polycarbonate 6 cm (2.35 inch) (5)
982165.003	Sample, polycarbonate 3.5 cm (1.4 inch) (5)
984308.001	Sample, 0.005 inch calibration
984308.002	Sample, 0.010 inch calibration
984308.003	Sample, 0.020 inch calibration
984308.004	Sample, 0.030 inch calibration
984308.005	Sample, 0.030 inch x.75 calibration
982166.003	Sample, .125 compliance, 6 cm (2.35 inch) long
982166.004	Sample, .125 compliance, 3.5 cm (1.4 inch) long
205221.002	Fuse, 10 Amp, 250 V
983169.001	Digital caliper B&S
270339.002	100 g weight
259508.000	Brass tweezers
280038.000	Tool, .25 hex head wrench, 3/32 bit
280039.000	Tool, 7/64 hex wrench, .25 bit
984347.001	Tool, 1/16 L hex wrench (mod)
280255.002	Tool, 9/64 L hex wrench, balldriver
280257.001	Tool, 3/32 L hex wrench, balldriver
984015.902	Dual cantilever bending stage
984015.903	Dual cantilever dovetail
270871.001	Socket setscrew, steel, .25 long cup point

Table 16: DMA Accessory Kit (985011.901) Items

Part Number	Description
204578.000	Lubricating spray
280041.001	Adapter, hex shank, 2-inch long with friction ball

**Table 17: Additional Replacement Parts** 

Part Number	Description
205221.001	Fuse (6.3 amp, 250 V)
251470.025	Ethernet cable (7.7m [25 foot], shielded)
253827.000	Power cord 120 V
985199.901	Control thermocouple (88.9 mm length)
985199.902	Sample thermocouple (127 mm length)
980228.902	Glass support cloth (0.205 mm thick, 32.92 m length)
984309.901	PET film samples 3.5 cm (1.5 inch) long (10)
984310.901	Indium wire samples 3.5 cm (1.5 inch) long (10)
984313.901	PET string sample 36" long
982165.904	Polycarbonate sample (0.794 mm thick, 35.56 mm long, pkg of 5)
984309.901	Polyethylene terephthalate (PET) film (pkg of 10 pieces, 38.1 mm long)
984313.901	PET fiber (914.4 mm long)
984054.001	Drive shaft
984003.901	DMA furnace
984370.901	Furnace inlet ferrule/spring kit
983164.001	Calibration sample thin film clamp
280257.001	Tool, wrench hex 3/32 L ball driver
280039.000	Hex wrench 7/64 0.25 bit
984347.001	Hex wrench 1/16 L
270962.001	Telescoping gauge (for 10 mm single cantilever clamps)
270962.002	Telescoping gauge (for 17 mm single cantilever clamps)
270976.002	Filter/regulator with auto-drain
270975.001	Filter element

**Table 17: Additional Replacement Parts** 

Part Number	Description
984350.901	Air compressor accessory
991400.902	Gas Cooling Accessory (GCA) - auto tank fill
984048.901	8 mm single/dual cantilever clamp kit
984047.901	20 mm singe/dual cantilever clamp kit
984015.901	35 mm single/dual cantilever clamp kit
984026.901	Three-point bending clamp kit (5, 10, and 15 mm lengths)
984014.901	Three-point bending clamp kit (20 and 50 mm lengths)
984018.901	Parallel plate compression clamp kit (includes 15 and 40 mm plates)
984022.901	Penetration kit
984016.901	Film/fiber tension clamp kit
985016.901	Dual surface film tension clamp
984023.901	Specialty fiber tension clamp kit
984017.901	Shear sandwich clamp kit
985067.901	Submersion compression clamp kit
985068.901	Submersion film/fiber clamp kit
985178.901	Three-point bending submersion clamp kit