# Q Series

# DMA-RH Accessory



**Getting Started Guide** 



#### **Notice**

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without 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:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- TA Instruments Offices

#### 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.

MISE EN GARDE: 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. 61010-1-04 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

CAN/CSA-22.2 No. 61010-2-010-04 Particular requirements for laboratory equipment for the heating of materials + Amendments.

#### For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN61010-1: 2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN61010-2-010: 2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

#### **For United States**

UL 61010-1: Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL 61010A-2-010 Part 2: Particular requirements for laboratory equipment for the heating of materials.

#### **Electromagnetic Compatibility Standards**

#### For Australia and New Zealand

AS/NZS CISPR11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

#### For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

#### For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 3 Protected EM environment.

#### 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.

#### **Accessory Symbols**

The following labels are displayed on the DMA-RH Accessory for your protection:

Symbol	Explanation	
<u>A</u>	This symbol indicates that you must unplug the accessory before doing any maintenance or repair work; AC power voltage is present in this system. High voltages are present in this accessory. 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 débrancher l'accessoire avant d'effectuer des travaux de maintenance ou de réparation ; présence d'une tension de secteur CA dans ce circuit. Présence de tensions élevées dans cet instrument/accessoire. Si vous n'êtes pas formé aux procédures électriques, ne déposez pas les couvercles de l'armoire sauf indications spécifiques contenues dans le manuel. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par le personnel d'entretien qualifié de TA Instruments.	
	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 *Q Series DMA-RH Accessory Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

#### Electrical Safety

You must unplug the accessory before doing any maintenance or repair work; voltages exceeding 120 VAC are present in this system



WARNING: High voltages are present in this accessory. 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.

AVERTISSEMENT: Présence de tensions élevées dans cet accessoire. Si vous n'êtes pas formé aux procédures électriques, ne déposez pas les couvercles de l'armoire sauf indications spécifiques contenues dans le manuel. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

#### Thermal Safety

CAUTION: After completing an experiment, allow the saturator valve and fitting (located on the back panel of the DMA-RH Accessory) to cool before touching.

MISE EN GARDE: À la fin de l'expérience, laissez la vanne et le raccord du saturateur (situés sur le panneau arrière de l'accessoire DMA-RH) refroidir avant de les toucher.

#### Chemical Safety



WARNING: Do not use hydrogen or any other explosive gas in the DMA-RH Accessory.

AVERTISSEMENT: N'utilisez pas d'hydrogène ou tout autre gaz explosif dans le accessoire DMA-RH.



WARNING: If analyzing samples that may emit harmful gases, vent the gases by placing the accessory near an exhaust.

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

### Lifting the Accessory



WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

AVERTISSEMENT: 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.

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# Chapter 1:

# Introducing the DMA-RH

### Overview

The DMA-RH Accessory is used with the TA Instruments DMA Q800 Dynamic Mechanical Analyzer. This accessory allows mechanical properties of a sample to be analyzed under controlled and/or varying conditions of both relative humidity and temperature. The DMA-RH Accessory is compatible with most Q800 clamps, with the specific exception of the submersion clamp. However, for best results, ensure that the sample has sufficient exposed surface area so that water vapor adsorption and desorption are not impeded. As such, the film/fiber, specialty fiber, cantilever, and three-point bend clamps are most appropriate for use with this accessory.

## System Components

The DMA-RH Accessory has the following major hardware components (see <u>Figure 1</u>):

- The DMA-RH Accessory cabinet precisely controls the temperature and humidity of the sample environment. The accessory cabinet is the main accessory component; it receives commands from the DMA via an RS-232 cable and controls all sub-systems that are associated with generating and maintaining both temperature and humidity.
- The heated transfer line is maintained at a temperature above the dew point temperature of the purge gas in order to avoid condensation.

• The sample chamber mounts on the DMA in place of the standard furnace and encloses the sample. Its temperature is controlled by the accessory cabinet, and it has been designed to accommodate most DMA clamps associated with DMA-RH experimentation.

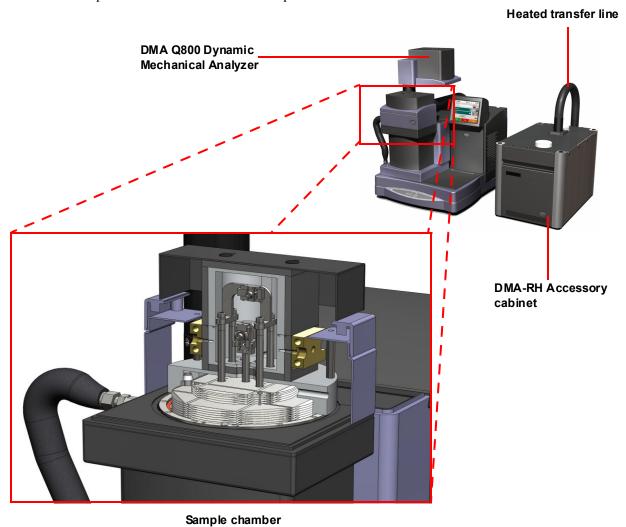


Figure 1 DMA-RH Accessory components; sample chamber cutaway.

# Accessory Specifications

The tables below detail the DMA-RH Accessory technical specifications.

**Table 1: DMA-RH Accessory Characteristics** 

Dimensions of control enclosure	Height: 14.5" Width: 12" Depth: 20"
Dimensions of sample chamber with attached transfer line	Height: 5 <sup>3</sup> / <sub>4</sub> " Width: 8 <sup>1</sup> / <sub>4</sub> " Depth: 7 <sup>1</sup> / <sub>4</sub> " Transfer line length: 7 feet
Weight of control enclosure	60 lbs (without water)
Weight of sample chamber with transfer line	7 lbs
Electrical inlet power requirements	Voltage: 90–265 VAC Frequency: 45–65Hz
Energy consumption	0.400 KVA

#### **Table 2: Accessory Gas Requirements**

Gas	Filtered nitrogen with dew point of –40°C or less	
Pressure	138 kPa gauge (20 PSI gauge)	
Volume	3 SLPM	

#### **Table 3: Accessory Environmental Conditions**

Operating temperature range	15°C to 35°C
Operating altitude	2000 meters maximum
Relative humidity	5% to 80% RH from 15°C to 31°C, decreasing to 66% RH at 35°C (non-condensing)

**Table 4: Accessory Performance Specifications** 

Temperature range	15°C to 35°C
Temperature accuracy	±0.5°C
Heating/Cooling rate	1°C/min
Humidity range	See <u>Figure 2</u> for the humidity range chart. Unless stated otherwise, specifications for humidity are at sea level. Units operated in higher elevations will have a reduced humidity operating range.
Humidity accuracy	5% to 90% RH, ±3% >90% RH, ±5%
Humidity ramp rate	2% RH/min (fixed), both increasing and decreasing (alternate RH pseudo-linear ramp rates can be achieved through programmed step-iso methods)

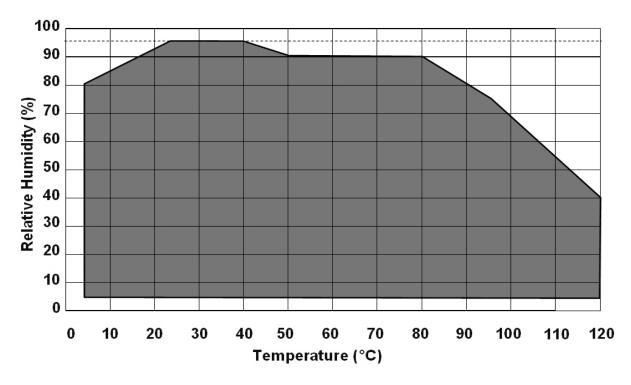


Figure 2 DMA-RH Accessory humidity range chart.

# Chapter 2:

# Installing the DMA-RH Accessory

## Unpacking/Repacking the DMA-RH

The instructions needed to unpack and repack the accessory are found as separate unpacking instructions in the shipping box and in the online documentation associated with the instrument control software. Be sure to read and perform the unpacking instructions prior to performing any procedures in this chapter.

Retain all of the shipping hardware and boxes from the accessory in the event you wish to repack and ship your accessory.



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.

## Preparing the Accessory

Before shipment, the DMA-RH 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 accessory for shipping damage and missing parts
- Connecting the DMA-RH cables and lines
- Filling the reservoirs
- Priming the Temperature Control System
- Filling the saturator
- Installing the sample chamber

It is recommended that you have your DMA-RH Accessory unpacked and installed by a TA Instruments Service Representative; call for an installation appointment when you receive your accessory.

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 the DMA-RH Accessory, look over the accessory and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

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

#### **Choosing a Location**

Choose a location for the accessory using the following guidelines. The DMA-RH Accessory should be:

#### In

- A temperature-controlled area. Temperatures should be in the range of 20–35°C.
- A clean environment, preferably on the ground floor in the building.
- An area with ample working and ventilation space.

#### On

A stable work surface.

#### Near

- A power outlet (universal input; 90–265 VAC, 47–63 Hz, 400 VA).
- Your TA Instruments thermal analysis controller.
- A nitrogen gas source and purge gas supplies with suitable regulators and filters, if required.
- Your Q800 DMA, but plan to place the DMA-RH Accessory cabinet on a separate table or on the floor.
- Be sure not to place the rear of the DMA-RH enclosure too close to a wall or other obstruction. The cooling fan is an integral part of the DMA-RH, and proper air flow is required for the operation of the system. Maintain at least 12 inches between the rear of the enclosure and any obstruction.

#### Away from

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Noisy or mechanical vibrations.
- High traffic areas, where constant movements from passing personnel could create air currents or mechanical disturbances.

### Connecting Cables and Lines

To connect the cables and gas lines, access the DMA-RH Accessory's rear panel and follow the instructions below:

CAUTION: When plugging in or unplugging power cords, handle them by the plugs, not the cord.

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

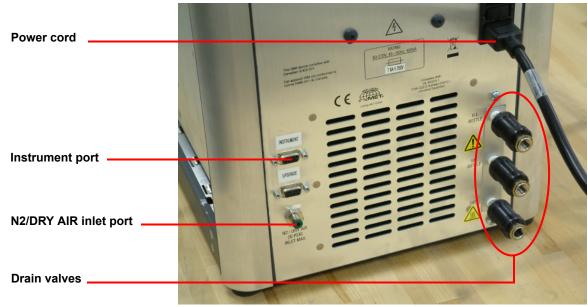


WARNING: Protect power and communications cable paths. Avoid laying cables across walkways, as this can create a tripping hazard.

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.

- 1 A note regarding the RS-232 communication cable:
  - If this is a first-time installation, do not yet connect the RS-232 communication cable. This will be done in subsequent steps.
  - If this is not a first-time installation (and you are sure there is no air in the water cooling lines), connect one end of the RS-232 cable to the INSTRUMENT port on the back of the DMA-RH and the other end of the cable to the COM2 port on the DMA instrument.
- 2 Connect the regulated nitrogen (15–20 psi) to the input of the supplied dryer, and connect the output of the dryer to the N2/DRY AIR inlet (shown below).
- 3 Connect the power cord to the DMA-RH Accessory, and plug the other end into the electrical outlet.

**NOTE**: Refer to <u>Q5000 SA Sorption Analyzer Getting Started Guide</u> for details on connecting lines to the dryer.



**Figure 3** Connection ports and drain valves on back of DMA-RH Accessory.

## Filling the DMA-RH Reservoirs

The Saturator Reservoir holds approximately 1 liter of distilled water. It can be filled at any time – even during an experiment – without adversely affecting data. The DMA-RH enclosure includes a micro-pump that automatically pumps distilled water from the Saturator Reservoir to the Saturator when it is low. It is important to maintain an adequate level of distilled water in the Saturator Reservoir to avoid an error. The Saturator Reservoir includes a liquid level sensor used to indicate a low liquid condition. If the LCD screen on the front of the DMA-RH enclosure reads "SAT RESERV LOW," fill the Saturator Reservoir with distilled water as soon as possible. An experiment will continue for some period of time (depending on experimental temperature and requested %RH) even when a low liquid condition exists. However, once the DMA-RH Accessory determines the water level in the Saturator is too low and no water is available in the Saturator Reservoir, the experiment will be terminated and the LCD screen will display "SAT WATER LOW."

The Heat Exchanger Reservoir also holds approximately 1 liter of distilled water, and supplies this as a coolant to the temperature control system. The temperatures of the Saturator and Sample Chamber are maintained by means of Peltier elements. The Peltier elements themselves are compensated by means of circulating water. The DMA-RH enclosure includes a circulating pump that moves water from the Heat Exchanger Reservoir, through the Peltier cooling blocks on the Saturator and Sample Chamber, and finally through a heat exchanger in the rear of the DMA-RH enclosure before returning it to the reservoir. It is important to maintain an adequate level of distilled water in the Heat Exchanger Reservoir to avoid termination of an experiment. The Heat Exchanger Reservoir includes a liquid level sensor used to indicate a low liquid condition. If the LCD screen on the front of the DMA-RH enclosure reads "COOLER LOW WATER", fill the Heat Exchanger Reservoir with distilled water as soon as possible. An experiment will continue even when a low liquid condition exists, as long as the system does not detect an over-temperature condition. If the water level in the Heat Exchanger Reservoir drops too low, it is possible to overheat the Peltier cooling blocks. An experiment will be terminated if the system detects an over-temperature condition.

- 1 Ensure that all connections in the previous section "Connecting Cables and Lines" are complete.
- 2 Ensure that all drain valves (circled in <u>Figure 3</u> above) are closed by pushing them in as far as allowable.
- Fill the HEAT EXCHANGER RESERVOIR and the SATURATOR RESERVOIR (located on the top panel of the DMA-RH Accessory; caps shown below) with distilled water.





Figure 4 HEAT EXCHANGER RESERVOIR (left); SATURATOR RESERVOIR (right).

CAUTION: Do not put any liquid other than distilled water into any of the DMA-RH Accessory's reservoirs.

MISE EN GARDE: Ne versez aucun liquide autre que de l'eau distillée dans les réservoirs de l'accessoire DMA-RH.

4. Power on the DMA-RH Accessory using the On/Off switch on the back panel of the accessory.

## Priming the Temperature Control System

The Temperature Control System must be purged of all air to ensure maximum efficiency of cooling and heating. This is accomplished using the Accessory Setup screen of the Humidity Accessory Test Application.

Prime the temperature control system upon initial installation, or if you suspect air has gotten into the lines.

- 1 Connect one end of the RS-232 communication cable to COM1 (or available serial port) on the PC. Connect the other end of the communication cable to the UPGRADE port on DMA-RH.
- 2 Navigate to C:\Program Files\TA Instruments\Thermal Advantage\Qseries\Test and then double-click the executable DMARH.exe to run the Humidity Accessory Application.
- 3 Click the Accessory Setup tab.
- In the **Select Serial Port** section, select the proper **COM** port from the drop-down list and click **Open Com Port**. Ensure that the status message **Successfully opened com port** displays in the **Status** window.

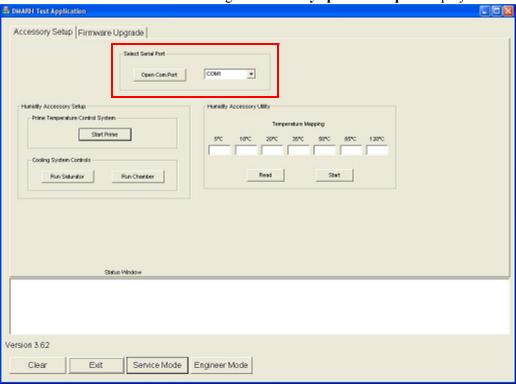
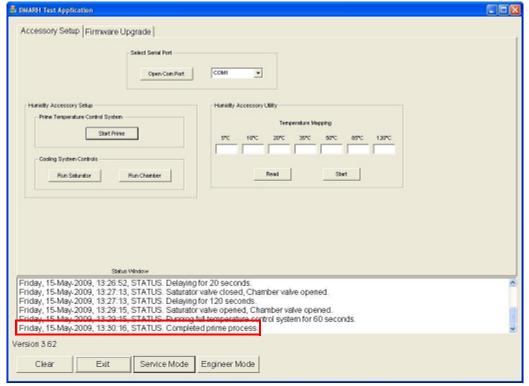


Figure 5 Open COM port.

- 5 Click **Start Prime** in the **Prime Temperature Control System** section. The DMA-RH begins the automated priming process, displaying each step in the **Status** window. At this time, the saturator pump will begin to click about two times per second, indicating that the pump is filling the saturator.
- When the process is complete, Completed prime process displays in the Status window, and water flow stops (a small amount of water may continue to flow).



**Figure 6** STATUS Completed prime process.

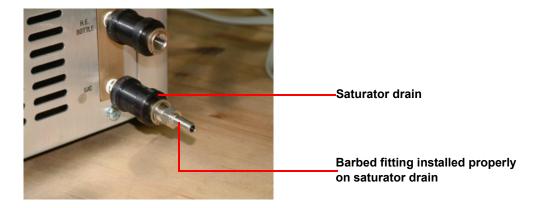
- 7 To ensure that the temperature control systems are properly primed, click **Run Saturator** in the **Cooling System Controls** section. Remove the lid from the Heat Exchanger bottle and verify water is flowing from the upper fitting into bottle. Click **Stop Saturator**.
- 8 Click **Run Chamber** and verify that water is flowing from upper fitting into Heat Exchanger bottle. Click **Stop Chamber**.

# Filling the Saturator

- 1 If necessary, disconnect the communication cable from the UPGRADE port on the DMA-RH and connect it to the INSTRUMENT port. Disconnect the other end from the PC and connect it to COM2 on the DMA instrument.
- 2 Install the barbed fitting (included in accessory kit and shown below in <u>Figure 7</u>) in the SAT (saturator drain) valve (see <u>Figure 8</u>). Hand-tighten the fitting.

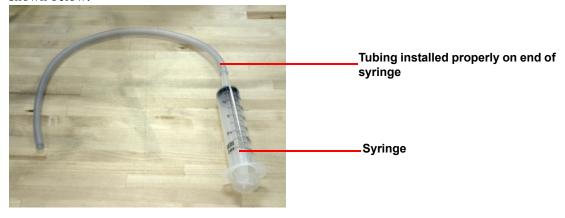


Figure 7 Saturator drain barbed fitting.



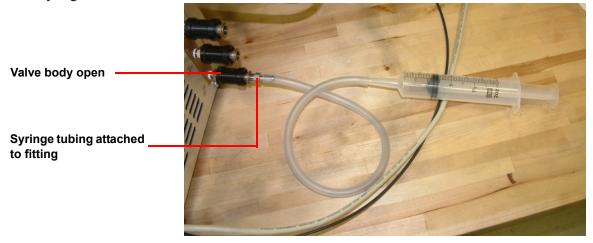
**Figure 8** Fitting installed properly in saturator drain.

3 Obtain the tubing and syringe from the accessory kit. Install the tubing onto the end of the syringe, as shown below.



**Figure 9** Proper syringe and tubing connections.

- 4 Fill the syringe with 60 cc of distilled water.
- 5 Connect the open end of the syringe tubing to the barbed fitting on the DMA-RH SAT valve.
- 6 Pull the valve body to open the valve and **slowly** dispense water into the DMA-RH Accessory by pushing in on the syringe.



**Figure 10** Filling the saturator through the saturator valve.

- 7 Close the saturator valve when the syringe empties and disconnect the syringe tubing from the barbed fitting.
- **8** Repeat step 4 through step 7 about two more times. The pump will continue to run (click) for about 15 minutes until the saturator is full.

CAUTION: Do not continue to add water if the pump stops clicking. It is possible to over-fill the saturator and push water into the sample chamber, potentially damaging the DMA.

MISE EN GARDE: Ne continuez pas à ajouter de l'eau si la pompe arrête de cliquer. Il est possible de remplir le tampon de saturation et de pousser l'eau dans la chambre à échantillon, ce qui pourrait endommager le DMA.

9 Remove the syringe tubing from the barbed fitting and, if desired, remove the fitting from the saturator valve. Again ensure that the saturator fitting valve is closed (pushed in).

CAUTION: If it becomes necessary to move the DMA-RH Accessory, it is necessary to drain some of the water out of the saturator. Failure to drain water from the saturator will result in water being pushed into the transfer line and then into the sample chamber when the purge gas is turned back on, potentially causing damage to the DMA. Follow the instructions given in "Moving the DMA-RH Accessory" on page 33.

MISE EN GARDE: S'il s'avère nécessaire de déplacer l'accessoire DMA-RH, il faut évacuer une partie de l'eau hors du tampon de saturation. Si vous n'évacuez pas l'eau du tampon de saturation, l'eau risque d'être poussée dans la conduite de transfert et ensuite dans la chambre à échantillon lorsque le gaz de drainage est remis en marche, ce qui pourrait endommager le DMA. Suivez les instructions fournies dans la section « Déplacement de l'accessoire DMA-RH » en page 33.

## Installing the DMA-RH Sample Chamber

To install the DMA-RH Accessory sample chamber onto the DMA Q800 Dynamic Mechanical Analyzer, follow the instructions below:

- 1 Open the furnace so that the DMA-RH sample chamber can be installed on the DMA.
- With two hands, carefully lift the furnace and continue to rotate it slightly toward the back of the DMA O800.
- 3 Remove any clamps that may be installed.
- 4 Remove the two mounting clips from the thermocouples with the hex wrench supplied in the accessory kit.
- 5 Insert the 1/8-inch hex wrench (provided in the kit) through the four holes in the sample chamber lid and loosen the captive screws until the lid can be removed.
- 6 Obtain the 1/8-inch tubing from the accessory kit, and install each end through the two small holes in the sample chamber where the thermocouples will pass through. Refer to <u>Figure 11</u> below for the proper location of the holes in the sample chamber.

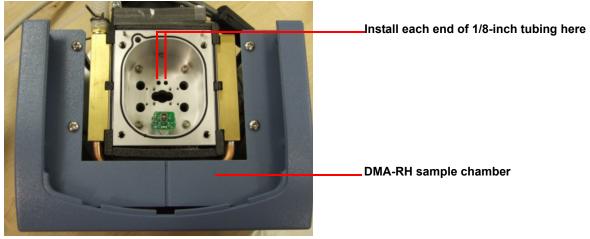


Figure 11 DMA-RH sample chamber and shaft cover.

7 Install the 4 mounting collars using the 4 lockdown bolts. Leave both the lockdown bolts and set screw loose.

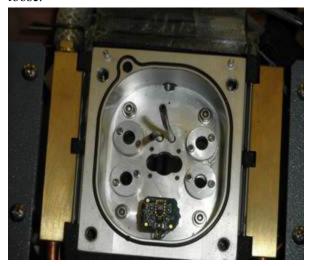


Figure 12 Mounting collars installed.

8 Hold the DMA-RH sample chamber over the DMA Q800 DMA sample area and place each thermocouple into each end of the 1/8-inch tubing, as shown below. Be careful not to break or damage either of the thermocouples' welded tips.

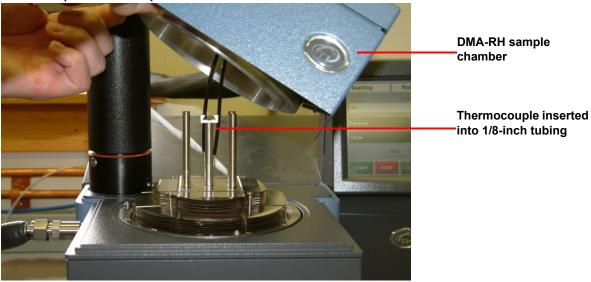


Figure 13 Installing the DMA-RH sample chamber on the DMA Q800 DMA cell.

9 Use the 1/8-inch tubing to guide the thermocouples through the two small holes in the DMA-RH sample chamber while simultaneously sliding the four clamp mounting posts through the matching four holes on the DMA-RH sample chamber. Be sure that the DMA-RH sample chamber sits securely on the DMA cell where the furnace would normally sit. When finished, remove the 1/8-inch tubing and place it back in the accessory kit for future use.

10 Make sure the DMA-RH sample chamber is sitting firmly on the DMA cell, then route the DMA-RH humidity control box transfer line around the back of the DMA cabinet, as shown below. Then attach the velcro strap around the handle and transfer line to prevent the DMA-RH sample chamber and transfer line from moving.

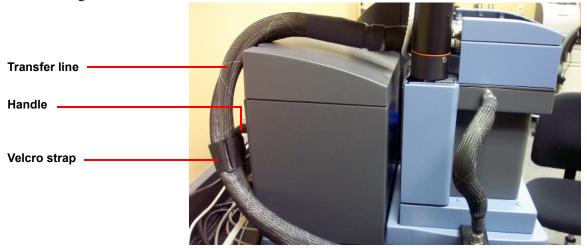


Figure 14 Transfer line routed properly.

11 Install the DMA shipping clamp onto the four posts (attaching to the drive shaft is not necessary). Finger-tighten the shipping clamp bolts.

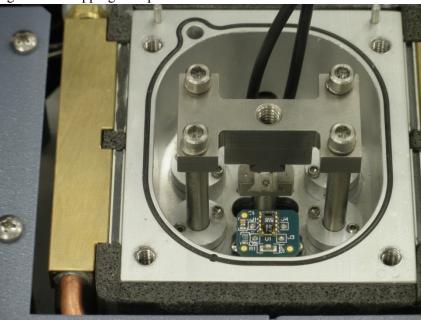


Figure 15 DMA-RH shipping clamp.

12 Working back and forth between the set screw and lockdown bolt, slowly tighten the lockdown bolt and then the corresponding set screw. Repeat for each post.

13 After all collars are tight, loosen the clamp bolts and remove the shipping clamp. Verify that the clamp can be easily removed and installed with the collars tight. If it cannot, loosen the collars and repeat step 9 after putting the shipping clamp back in place.



Figure 16 Split collars.

- 14 Install the two halves of the shaft collar. Position the collars to minimize the gap between the collars and the drive shaft, as shown below.
- 15 Place the shaft collar retaining ring into position on the drive shaft collar, as shown below.

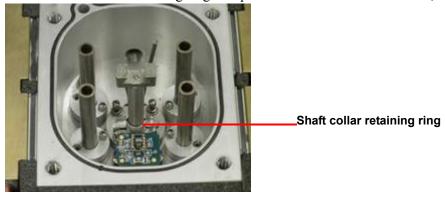


Figure 17 Shaft collar retaining ring in position on drive shaft collar.

16 Perform a slide motion test to verify that the drive shaft collars are not rubbing on the drive shaft.

17 Install the two thermocouple mounting clips to hold the thermocouples in place. These clips should also come in contact with the bottom of the sample clamp mounting posts. Refer to the figure below.



Figure 18 DMA-RH sample chamber installed properly.

**18** Install and calibrate the desired DMA sample clamp.

**NOTE**: Submersion clamps will not work when the DMA-RH sample chamber is installed. In addition, the standard compression, miniature three-point bend, and penetration clamps are also not compatible with the DMA-RH system. A specialty kit is available which contains new DMA-RH specific compression, penetration, and miniature three-point bending clamps for use with the DMA-RH Accessory.

19 Install the sample chamber lid and use the 1/8-inch hex wrench in the kit to tighten the four captive screws before running an experiment.

# Setting up the DMA-RH Accessory using Instrument Control Software

After successfully installing the DMA-RH Accessory, access the DMA Thermal Advantage Software and follow the instructions below.

- 1 Select Tools > Instrument Preferences.
- 2 Select the **DMA** tab.
- 3 Select the **Humidity chamber** check box.
- 4 Click **OK** to enable control of the DMA-RH Accessory.

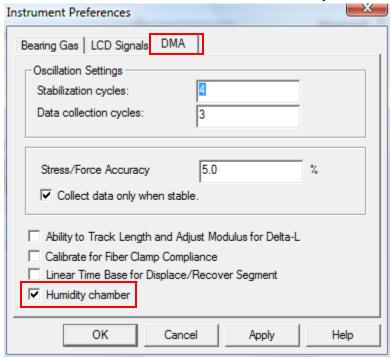


Figure 19 Instrument Preferences.

# Chapter 3:

# Use, Maintenance, & Diagnostics

# Using the DMA-RH Accessory

All of your DMA-RH Accessory experiments will follow the same 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. See the instrument control online help for instructions on performing these actions.

- Filling the saturator reservoir
- Selecting and preparing the sample
- Creating or choosing a test procedure and entering experiment information through the TA Instruments instrument control software
- Loading the prepared sample
- Starting the experiment

#### **Before You Begin**

Before using the DMA-RH Accessory, ensure that the DMA Q800 Dynamic Mechanical Analyzer is installed properly. Also make sure you have:

- Connected the RS-232 cable between the DMA Q800 Dynamic Mechanical Analyzer and the DMA-RH Accessory
- Connected enclosure power line and gas line
- Powered on each unit
- Filled the reservoirs as necessary
- Specified the DMA-RH Accessory in the instrument control software
- Become familiar with controller operations

### Running a DMA-RH Experiment

#### **Loading the Sample**

If necessary, connect the communication cable from the INSTRUMENT port on the DMA-RH to the COM2 port on the DMA instrument.

See online help for details concerning sample preparation and loading. The same general guidelines are applicable as a standard DMA experiment.

#### **Creating an Experimental Procedure**

The Q800 DMA instrument control software allows a variety of sorption analysis experiments to be run, including stepped humidity at constant temperature and stepped temperature while holding humidity constant.

See online help for details concerning creating and running a method. The same general guidelines are applicable as a standard DMA experiment.

After enabling control of the DMA-RH (see "<u>Setting up the DMA-RH Accessory using Instrument Control Software</u>" on page 27), the following three method segments become available from the **Segment** list (shown below).

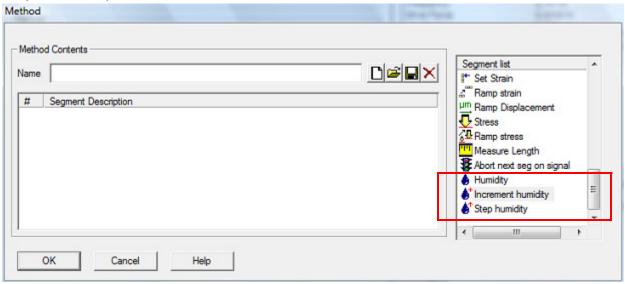


Figure 20 Segment list.

• **Humidity**: The **Humidity** command allows you to specify a static relative humidity for the instrument to maintain. It is often used to program the initial relative humidity for an experiment. Upon starting the experiment, the DMA-RH ramps to the selected humidity at a set rate of 2% RH/min.

When using the **Humidity** segment, the running method advances to the next segment before it attains the specified humidity. Therefore, it is necessary to follow this **Humidity** segment with an **Isothermal** segment. When specifying the time for the **Isothermal** segment, include the time it will take to reach your specified humidity (at a rate of 2% RH/min), plus the time you want to wait for the system to reach equilibrium after the humidity has been achieved.

- **Increment humidity**: The **Increment Humidity** segment raises or lowers the relative humidity in a controlled step, then begins the next segment. It is usually followed by an **Isothermal** segment for the same reason the Humidity segment is followed by an **Isothermal** segment (as explained above). This method segment is often used in conjunction with the Repeat segment to construct a series of humidity steps over a specified range.
- Step humidity: The Step Humidity segment causes the relative humidity to jump a specified increment at a specified time interval until a final relative humidity is reached (the relative humidity can increase or decrease at a maximum of 2% RH/min, regardless of the programmed step/interval). This segment automatically turns on data collection, except when preceded by a **Data Storage OFF** segment. Enter the percent increment, the length of time (in minutes) you wish to hold at this step, and the final humidity set point. The length of time to hold at each step must include the amount of time it will take to reach that step (at a rate of 2% RH/min), plus the amount of time you wish to wait for the system to reach equilibrium once the humidity has been achieved.

Other factors related to method segments to take into consideration when using the DMA-RH:

- It is important to note that while in Standby mode, the DMA-RH Accessory purges the sample chamber with dry gas (0% RH). This is a safety feature and cannot be changed. If you prefer to keep the humidity level at a condition other than 0% RH, you must program that condition as the last step in an experiment, and keep the method active between runs.
- In DMA-RH mode, the temperature **Equilibrate** segment acts similar to a temperature **Jump** segment in that the software does not wait for stabilization criteria. During an **Equilibrate** segment, the Q800 DMA passes the requested temperature to the DMA-RH accessory and allows the accessory to control the temperature of the sample chamber. The temperature of the sample chamber is driven to a value close to the requested value (typically within 5°C), and the Q800 DMA software then steps to the next segment in the method. The DMA-RH accessory eventually drives the temperature to the requested value, but it is necessary to place an **Isothermal** segment after every **Equilibrate** segment in order to give the system enough time to stabilize. Alternatively, use a **Ramp** temperature segment can be used. However, an **Isothermal** segment is needed after every **Ramp** segment as well.
- The **Motor Drive** method segment is typically used to lock and unlock the drive shaft at various times during an experiment, halting mechanical inputs to the sample. However, it is necessary to maintain the purge gas from the air bearings during DMA-RH experiments. Therefore, while in DMA-RH mode, the **Motor Drive OFF** segment allows the shaft to float rather than lock. All mechanical inputs to the sample still turn off. When setting the motor drive to OFF, you must first set the relative humidity to 0%, otherwise the experiment will be terminated. Therefore, the order of segments must be **Humidity 0%**, then **Motor Drive OFF**.

## Maintaining the Accessory

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.



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

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

#### **Draining the DMA-RH Reservoirs**

The procedure below explains how to properly drain the DMA-RH Reservoirs.

- 1 Turn off the power switch on the accessory and unplug the power cord from the power inlet. Remove the power supply side panel (the right side panel if facing the drain valves).
- 2 Remove both the Heat Exchange Reservoir and Saturator Reservoir caps.
- 3 Install the barbed fitting (included in accessory kit and shown in <u>Figure 7</u>) in the SAT (saturator drain) valve (refer to Figure 8).
- 4 Obtain the tubing from the accessory kit. Install the tubing onto the end of the barbed fitting, and pull the SAT valve open to drain. Use a bucket or a similar apparatus to catch the draining liquid.
- 5 Once the draining is complete, remove the barbed fitting and tubing from the SAT drain valve.
- 6 Attach the fitting and tubing to the SAT BOTTLE drain valve, and pull the SAT BOTTLE valve open to drain. Again, use a bucket or a similar apparatus to catch the draining liquid.
- 7 Once the draining is complete, remove the barbed fitting and tubing from the SAT BOTTLE drain valve.
- **8** Attach the fitting and tubing to the H.E. BOTTLE drain valve. Pull the H.E. BOTTLE drain valve open to drain, using a bucket or similar apparatus to catch the draining liquid.

9 Pinch off supply line from the Heat Exchange reservoir above the "tee" fitting using the hemostat pliers supplied in the accessory kit. Refer to the figure below.

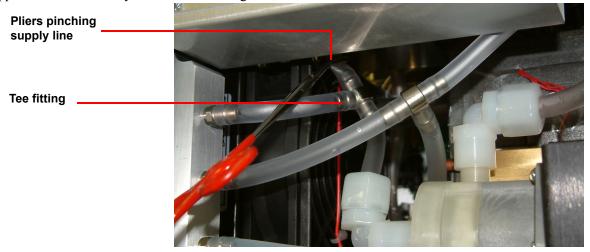


Figure 21 Hemostat pliers pinching off supply line, just above "tee" fitting.

- 10 Obtain the syringe from the accessory kit and retract the plunger. Attach the syringe to the tubing.
- 11 Push the plunger into the syringe, forcing water from the cooling system back into the Heat Exchange reservoir.
- 12 Close the drain valve, remove the tubing (with syringe still attached) from the fitting, and retract the plunger.
- 13 Reattach the tubing and syringe to the fitting and open the drain valve. Push the plunger into the syringe.
- 14 Repeat step 12 and step 13 (approximately 15 times) until all water is forced from the cooling system.
- 15 When all water is forced from the cooling system, close the drain valve, remove the hemostat pliers pinching off the supply line, and remove the syringe from the tubing. Then open the H.E. BOTTLE drain valve to drain the remaining water from the H.E. Reservoir, using a bucket or similar apparatus to catch the draining liquid.
- 16 When finished, replace the reservoir caps and reattach the power supply side panel.

### **Cleaning the Accessory**

To clean the DMA-RH Accessory, wipe down the control cabinet with a damp, soft cloth.

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

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

#### **Moving the DMA-RH Accessory**

CAUTION: If it becomes necessary to move the DMA-RH Accessory, it is important to drain some of the water out of the saturator. Failure to drain water from the saturator will result in water being pushed into the transfer line and then into the sample chamber when the purge gas is turned back on, causing potential damage to the DMA.

MISE EN GARDE: S'il s'avère nécessaire de déplacer l'accessoire DMA-RH, il faut évacuer une partie de l'eau hors du tampon de saturation. Si vous n'évacuez pas l'eau du tampon de saturation, l'eau risque d'être poussée dans la conduite de transfert et ensuite dans la chambre à échantillon lorsque le gaz de drainage est remis en marche, ce qui pourrait endommager le DMA.

Follow these steps to drain a small amount of water from the saturator:

- 1 Turn off the power switch on the accessory and unplug the power cord from the power inlet.
- 2 Obtain a bucket or small container to collect the water that will be drained from the saturator.
- 3 Locate the saturator drain fitting (shown in <u>Figure 7</u>) and pull the valve body to open the valve and allow water to flow into the container. Drain at least 100 mL of water from the saturator prior to moving the DMA-RH.
- 4 Move the DMA-RH to a new location, following the guidelines found in <u>"Choosing a Location" on page 15</u>in this manual.
- 5 Reattach all cables and lines per "Connecting Cables and Lines" on page 16 found in this guide.

#### **Downloading Firmware to the DMA-RH Accessory**

Upon delivery, the latest firmware is already installed on the DMA-RH. However, in the event that you need to update the firmware, follow the procedure below.

- 1 Attach a RS-232 cable to the serial port on the controller and to the serial port labeled UPGRADE on the rear of the DMA-RH Accessory.
- 2 Navigate to the directory C:\Program Files\TA Instruments\Thermal Advantage\LDR.
- 3 Double-click on the executable file **DMARH.exe** to open the DMAR-RH Accessory Application.
- 4 When the application opens, select the **Firmware Upgrade** tab.



Figure 22 Firmware Upgrade tab.

5 Verify that the Flash Program File Selector is set to the following path: C:\Program Files\TA Instruments\Thermal Advantage\Qseries\InstrumentSW\DMA\QNX\HUMIDITY\DMARH.xxx

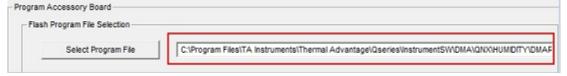


Figure 23 Verify path.

- 6 If the Flash Program File Selector is set to a different path, click Select Program File and navigate to the path listed in step 5.
- 7 Under **Firmware Program Control**, click the drop-down menu to the right of **Open Com Port**. Select the RS-232 Com Port that the cable is connected to on the controller. Then click **Open Com Port**.



A message similar to the one below displays.

Tuesday, 12-May-2009, 14:10:36, STATUS. Open serial port successfully

Figure 24 Status message.

8 Click Firmware Version to obtain the current version of firmware installed on the DMA-RH Accessory.



Figure 25 Firmware Version.

9 Click **Upgrade Programmed Board** to start the firmware download process.

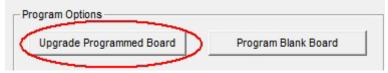


Figure 26 Upgrade Programmed Board.

A message similar to the one below displays.

Tuesday, 12-May-2009, 14:16:37, STATUS. Starting firmware download process. Tuesday, 12-May-2009, 14:16:39, STATUS. Downloading firmware to Accessory.

Figure 27 Status message.

10 When the firmware download completes, a status message similar to the one below displays within the DMA-RH Accessory application.

Figure 28 Status message.

- 11 After receiving the message that the firmware was successfully downloaded to the DMA-RH Accessory, click **Exit** to close the application.
- 12 As part of the download process, the DMA-RH Accessory automatically reboots.
- 13 Disconnect the RS-232 communication cable between the controller and UPGRADE port on the rear of the DMA-RH Accessory.

# Mapping Temperature on the DMA-RH Accessory

If the RH sensor is replaced, it is necessary to map the new sensor's response to the sample chamber. Additionally, if the measured temperature drifts away from the requested temperature by an unacceptable amount, perform the temperature mapping process as a means to reconcile the values. Follow the procedure below to map the temperature sensor:

- 1 Attach an RS-232 cable to the serial port on the controller and to the serial port labeled INSTRUMENT on the rear of the DMA-RH Accessory.
- 2 Navigate to the directory C:\Program Files\TA Instruments\Thermal Advantage\LDR.
- 3 Double-click on the executable file **DMARH.exe** to open the DMAR-RH Accessory Application.
- 4 When the application opens, click the **Accessory Setup** tab.



Figure 29 Accessory Setup tab.

5 Click the drop-down menu to the right of **Open Com Port**. Select the RS-232 Com Port that the cable is connected to on the controller. Then click **Open Com Port**.



Figure 30 Select Serial Port.

6 Click **Start** to begin the temperature mapping process. Note that this process will take approximately 8 hours to complete.



Figure 31 Humidity Accessory Utility.

7 Upon completion of the temperature mapping, the values appear in the boxes, and the DMA-RH Accessory automatically stores the values in flash memory.

## Monitoring the Humidity Chamber

The water reservoir within the DMA-RH enclosure will require periodic refills with distilled water. When the reservoir is low, a message will appear in the status line. Follow the instructions on page 16 to fill the chamber again.

**NOTE**: The "SAT RESERV LOW" indicator initially triggers when approximately 250 mL are left in the reservoir.

The water reservoir in the DMA-RH holds approximately 1 L of water when full. The rate of consumption of that water during experiments is dependent on temperature, %RH requested, and time. The dominant factor, however, is temperature. At 25°C, the rate of consumption is low, even if high humidities are used. At 25°C it takes more than 30 days for the reservoir to be depleted. On the other hand, at extreme conditions (for example, isothermal at 85°C and 85% RH), the rate of water consumption is much higher.

**NOTE**: It is good practice to always fill the water reservoir prior to starting an experiment. Water can be added in the middle of an experiment with no adverse affects to the data.

# 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-RH contains internal fuses that are not user serviceable. If any of the internal fuses blow, a hazard may exist. Call your TA Instruments service representative.

The only fuses that you can replace yourself are the fuses located in the power entry module located at the rear of the accessory. To check or change these fuses:

- 1 Turn the accessory off and remove the power cord.
- 2 Remove the fuse drawer from the accessory by using your finger to pry out the edge of the drawer, then removing the entire fuse drawer (see <u>Figure 32</u>).

CAUTION: Do not use a metal tool to remove the fuse drawer from the DMA-RH Accessory, as it could result to damage to the fuse and/or fuse drawer.

MISE EN GARDE: N'utilisez pas un outil métallique pour retirer le tiroir à fusible de l'accessoire DMA-RH, car cela pourrait endommager le fusible et/ou le tiroir à fusibles.

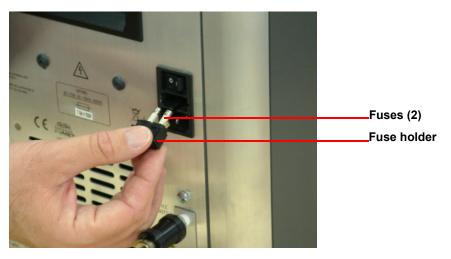


Figure 32 Fuses on DMA-RH Accessory.

- 3 Remove old fuses and replace the fuses only with the type and rating indicated on the accessory's rear panel.
- 4 Place fuse holder back into opening and push until it sits securely in the slot.

# Replacement Parts

**Table 5: Replacement Parts for the DMA-RH Accessory** 

Part Number	Description
985606.901	Mass flow control assembly
985670.901	DMA RH sensor PC board
985680.901	DMA RH control PC board
985723.901	Post collar assembly
985703.901	Shaft collar assembly

# Error Messages and Troubleshooting

The following are error codes that display in the software and on the DMA touch screen; included in this list are possible troubleshooting solutions for each error code.

LCD Display Text	Instrument & Controller Display Text	Try the Following
Bad Press Sensor	Error 350. Bad pressure sensor. Call TA Instruments.	Reboot the DMA-RH box. If problem persists, call TA Instruments.
Encl Over Temp	Error 351. DMA-RH Accessory enclosure temperature too high. Call TA Instruments.	Turn the DMA-RH off, and let sit for several minutes. Try powering it back on and running another experiment. If problem persists, call TA Instruments.
No Instrum Comm	Error 352. Communication time out with DMA-RH accessory.	Check the RS-232 cable connections at both the instrument and the DMA-RH. The cable should be connected to the COM2 port on the instrument, and the INSTRUMENT port on the DMA-RH.
Low 24V Power	Error 353. Power Supply. Low 24V. Call TA Instruments.	Call TA Instruments.
No Mass Flow Cal	Error 354. No calibration parameters found for Mass Flow Controllers.	Power cycle the DMA-RH. If the error persists, call TA Instruments.
MFC Checksum Err	Error 355. Error reading calibration parameters for Mass Flow Controllers.	Turn off the power to the DMA-RH. Wait several minutes, then turn the power back on. If the error persists, call TA Instruments.
MFC 100% Sensor	Error 356. Unable to attain purge gas flow. Call TA Instruments.	Ensure a dry gas purge line is connected to the rear of the DMA-RH. If problem persists, call TA Instruments.
MFC 0% Sensor	Error 357. Unable to attain purge gas flow. Call TA Instruments.	Ensure a dry gas purge line is connected to the rear of the DMA-RH. If problem persists, call TA Instruments.
MFC 100% Balance	Error 358. Problem controlling humidity to proper level. Damage to DMA could be possible. Call TA Instruments as soon as possible.	Call TA Instruments.
MFC 0% Balance	Error 359. Problem controlling humidity to proper level. Damage to DMA could be possible. Call TA Instruments as soon as possible.	Call TA Instruments.

Sat Water Low	Error 360. Saturator water level is too low, and saturator reservoir bottle is empty. Refill saturator reservoir bottle.	Fill the saturator reservoir bottle near the front of the DMA-RH with distilled water. See "Filling the DMA-RH Reservoirs" on page 17 in this manual for help. To clear the fault, turn the DMA-RH off and then back on again. A clicking sound will be heard as the system refills the saturator to its proper level.
Sat Refill Error	Error 361. Error refilling saturator. Call TA Instruments.	Call TA Instruments.
Sat Heat Sink OT	Error 362. Saturator heat sink temperature too high. Call TA Instruments.	In this fault state, the DMA-RH should continue to cool itself and to bring the temperature down to a safe level. Leave the DMA-RH alone for several minutes to allow it to cool sufficiently. The LCD screen on the DMA-RH will continue to post this message, even if the system is able to cool the heat sink sufficiently. After cooling down, a new experiment can be started. If the system is able to run, it will clear the message and enter operational mode. If the system cannot run, and continues to post this error message, call TA Instruments.
Sat Wall Temper	Error 363. Saturator temperature too high. Call TA Instruments.	In this fault state, the DMA-RH should continue to cool itself and to bring the temperature down to a safe level. Leave the DMA-RH alone for several minutes to allow it to cool sufficiently. The LCD screen on the DMA-RH will continue to post this message, even if the system is able to cool the saturator sufficiently. After cooling down, a new experiment can be started. If the system is able to run, it will clear the message and enter operational mode. If the system cannot run, and continues to post this error message, call TA Instruments.
No Humidity Cal	Error 365. No calibration parameters found for humidity.	Turn off the power to the DMA-RH. Wait several minutes, then turn the power back on. If the error persists, call TA Instruments.
Sat Cal Wrt Err	Error 366. Error writing humidity calibration values to memory.	Turn off the power to the DMA-RH. Wait several minutes, then turn the power back on. If the error persists, call TA Instruments.
Sat Cal Read Err	Error 367. Error reading humidity calibration values from memory.	Turn off the power to the DMA-RH. Wait several minutes, then turn the power back on. If the error persists, call TA Instruments.

Sat Reserv Low	Error 369. Saturator reservoir water level is low. Refill the saturator reservoir as soon as possible.	Refill the saturator reservoir bottle toward the front of the DMA-RH with distilled water. See "Filling the DMA-RH Reservoirs" on page 17 in this manual for assistance.
Cham Sensor Open	Error 370. Error reading Sample Chamber humidity and temperature sensor. Call TA Instruments.	Turn off the power to the DMA-RH. Wait several minutes, then turn the power back on. If the error persists, call TA Instruments.
Cham High Dew Pt	Error 371. Dew Point Temperature in Sample Chamber is too high. Run ended.	In this state, the system has detected a dew point temperature that could cause condensation to form within the sample chamber. As a safety feature, the system stops the run and purges the sample chamber with dry gas. Allow the DMA-RH to remain in standby mode for several minutes to allow the system to purge the sample chamber. After several minutes it is safe to begin the experiment again.
Cham Heat SNK OT	Error 372. Sample Chamber heat sink temperature too high. Call TA Instruments.	In this fault state, the DMA-RH should continue to cool itself and to bring the temperature down to a safe level. Leave the DMA-RH alone for several minutes to allow it to cool sufficiently. The LCD screen on the DMA-RH will continue to post this message, even if the system is able to cool the heat sink sufficiently. After cooling down, a new experiment can be started. If the system is able to run, it will clear the message and enter operational mode. If the system cannot run, and continues to post this message, call TA Instruments.
Chamb Over Temp	Error 373. Sample Chamber temperature too high. Call TA Instruments.	Call TA Instruments.
Xfer High Temp	Error 374. Transfer line temperature too high. Call TA Instruments.	Call TA Instruments.
Xfer High Dew Pt	Error 375. Dew Point temperature of purge gas is higher than transfer line temperature. Restart run.	The system detected a low temperature condition in the transfer line and entered into standby mode. Allow the system to remain in standby mode for a few minutes to allow dry gas to purge through the line. Restart run.
Cooler Low Water	Error 376. Heat Exchanger reservoir water level is low. Refill the heat exchanger reservoir as soon as possible.	Refill the heat exchanger reservoir bottle toward the back of the DMA-RH with distilled water. See "Filling the DMA-RH Reservoirs" on page 17 in this manual for assistance.

Setting Truncate	Error 378. A temperature or humidity request is outside of the operational range of the DMA-RH. The setting was truncated to the closest valid setting.	This typically occurs when the DMA-RH receives a humidity setpoint higher than its established operational range. In this case, the DMA-RH will control the humidity to the highest valid setting within its operational range. The run will continue normally, and all data will be saved.
Chamb Temp Delta	Error 379. Temperature delta between the Sample Temperature sensor and the Sample Chamber wall is too high. Run ended.	This typically occurs if the DMA-RH begins a run while the sample chamber top is not installed, or if the sample chamber top is removed during an experiment. Ensure the sample chamber top is installed properly, and then restart the run. If the problem persists, call TA Instruments.
Humidity Backoff	Error 380. The humidity level in the Sample Chamber was reduced slightly to avoid condensation on the chamber wall.	This condition occurs if the system detects the temperature of the sample chamber wall has fallen below the dew point temperature of the environment within the sample chamber. The system will dynamically control the humidity level down slightly to ensure no condensation occurs within the sample chamber. The run will continue normally, and all data will be saved.

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