

**TA** *Instruments*

Thermal Analysis & Rheology  
A SUBSIDIARY OF WATERS CORPORATION



# *Sample/ Temperature Stage*

*Accessory for the  $\mu$ TA 2990<sup>TM</sup>*

*Operator's Guide*

PN 899038.001 Rev. A  
Issued July 2000

© 2000 by TA Instruments  
109 Lukens Drive  
New Castle, DE 19720

### **Notice**

The material contained in this manual is believed adequate for the intended use of this 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. This publication is not a license to operate under or a recommendation to infringe upon any process patents.

TA Instruments Instrument Control, Data Analysis, and Utility Software and their associated manuals are proprietary and copyrighted by TA Instruments, Inc. Purchasers are granted a license to use these software programs on the instrument and controller with which they were purchased. These programs may not be duplicated by the purchaser without the prior written consent of TA Instruments. Each licensed program shall remain the exclusive property of TA Instruments, and no rights or licenses are granted to the purchaser other than as specified above.

---

# Table of Contents

Hotlines to TA Instruments .....	v
Part Numbers .....	vii
Notes, Cautions and Warnings .....	viii
Safety .....	ix
High Voltage .....	ix
Safe Handling of Cryogenic Materials .....	x
Handling Liquid Nitrogen .....	x
If Burned by Liquid Nitrogen .....	xi
Room Ventilation .....	xi
Oxygen Absorption .....	xi
Instrument Symbols .....	xiii
Introducing the Sample/ Temperature Stage .....	1-1
Description .....	1-2
Specifications .....	1-4
Principles of Operation .....	1-5
Installing the Sample/ Temperature Stage .....	1-7
Choosing a Location .....	1-7
Power Requirements .....	1-7
Unpacking and Assembly .....	1-8
Installing the Sample Cover .....	1-11
Filling the Cooling Canister .....	1-13
Operating the Sample/ Temperature Stage .....	1-15
Basic Procedure .....	1-15
Using the Stage Controller .....	1-17
Setting the Rate .....	1-18

<b>Selecting the</b>	
Target Temperature .....	1-20
Using the Start/Finish Button .....	1-21
Using the Hold Button .....	1-22
Using the Heat/Cool Button .....	1-23
Sample Procedure .....	1-24
<b>Performing Temperature Calibration .....</b>	<b>1-25</b>
<b>Removing the</b>	
Old Calibration Factors .....	1-25
<b>Obtaining New Positive</b>	
Calibration Factors .....	1-26
<b>Entering the New</b>	
Calibration Factors .....	1-27

---

## Hotlines to TA Instruments

TA Instruments, Inc.  
109 Lukens Drive  
New Castle, DE 19720  
Telephone: 1-302-427-4000 or 1-302-427-4040  
Fax: 1-302-427-4001

Email address: <http://www.tainst.com>; click on “Answerman” icon.

HELPLINE—U.S.A.: For technical assistance with current or potential thermal analysis applications, phone the Thermal Analysis Help Desk at (302) 427-4070.

SERVICE—U.S.A.: For instrument service and repairs, phone (302) 427-4050.

TA Instruments Ltd.  
Europe House, Bilton Centre  
Cleeve Road  
Leatherhead, Surrey KT22 7UQ  
England  
Telephone: 44-1-372-360363  
Fax: 44-1-372-360135

TA Instruments Spain  
Waters Cromatografía, S.A.  
División TA Instruments  
Avda. Europa, 21. Pta. Baja  
28108 Alcobendas  
Madrid  
Spain  
Telephone: 34-91-661-8448  
Fax: 34-91-661-0855

TA Instruments France  
B.P. 608  
78056 Saint-Quentin-Yvelines  
Cedex  
France  
Telephone: 33-1-30-48 94 60  
Fax: 33-1-30-48 94 51

TA Instruments GmbH  
Max-Planck-Strasse 11  
D-63755 Alzenau  
Germany  
Telephone: 49-6023-9647-0  
Fax: 49-6023-9647-77

TA Instruments Belgium, a Division of Waters  
s.a/n.v.  
Raketstraat 60  
B-1130 Brussels  
Telephone: 32-2- 706 00 80  
Fax: 32-2- 706 00 81

TA Instruments The Netherlands  
A Division of Waters Chromatography B.V.  
Florijnstraat 19  
4879 AH Etten-Leur  
Telephone: 31-76- 508 72 70  
Fax: 31-76- 508 72 80

TA Instruments, Japan  
No. 5 Koike Bldg.  
1-3-12 Kitashinagawa  
Shinagawa-Ku, Tokyo 140  
Japan  
Telephone: 813/3450-0981  
Fax: 813/3450-1322

TA Instruments Australia  
Unit 3  
38-46 South Street  
Rydalmere NSW 2116  
Australia  
Telephone: 61-29-9331-705  
Fax: 61-29-8981-455

TA Instruments Italy  
Division of Waters SpA  
via Achille Grandi 27  
20090 Vimodrone (MI)  
Italy  
Telephone: 39-02-27421-1  
Fax: 39-02-250-1827

---

## Part Numbers

If you need to order a replacement part for your Sample/Temperature Stage, refer to the following list of parts and then contact your service representative or the TA Instruments office closest to you. A list of the offices can be found on page v.

Part Number	Description
899037.001	Funnel
899036.001	Sample Cover
899038.001	Manual Sample/Temperature Stage
271446.001	Spacer

## Notes, Cautions and Warnings

The following conventions are used throughout this manual to point out items of importance and safety as you read through the instructions.

**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.



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

---

## Safety

Please read the safety information contained in this section carefully *BEFORE* using the instrument. The knowledge that you gain could save you from injury or equipment damage as you use the Sample/Temperature Stage.

### *High Voltage*



**Wherever high voltage is present on the system, extreme care should always be taken to avoid direct contact while the instrument hardware is powered on. Call TA Instruments Service for repairs. We do not recommend that you make any attempt to remove the panels on the instrument. If you do attempt your own repairs, always turn the power off before attempting to remove any panels or PC boards and before touching any connectors by hand or with electrically conductive tools. High voltage is present at the following locations on the**

**system and where the  symbol is located:**

- **Power/connection cables: Temperature Stage and Controller.**

## Safety

*(continued)*

### *Safe Handling of Cryogenic Materials*

Liquid nitrogen is used as a cooling agent in many thermal analysis tests. Because of its extremely low temperature ( $-196^{\circ}\text{C}$ ), it will burn the skin. You **must** use extreme care when working with liquid nitrogen or other cryogenic materials.

Liquid nitrogen can:

1. Cause serious skin burns.
2. Replace the air in the room you are in.
3. Generate very high pressures if trapped in lines or containers.

### *Handling Liquid Nitrogen*

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 shoe tops.
2. Transfer the liquid slowly to prevent thermal shock to the container and excessive turbulence to the liquid nitrogen. If liquid nitrogen is poured into a container that is at room temperature, the liquid nitrogen will boil violently. Use extreme caution to prevent the boiling liquid from contacting you.

---

## Safety

*(continued)*

3. Use only approved low-temperature containers.
4. Make sure liquid nitrogen containers are vented to prevent pressure buildup.

### *If Burned by Liquid Nitrogen*

- |  |
|--|
| <ol style="list-style-type: none"><li>1. Flood the area (skin or eyes) IMMEDIATELY with large quantities of cool water, and then apply cold compresses.</li><li>2. See a doctor IMMEDIATELY if the skin is blistered or if the liquid nitrogen came in contact with your eyes.</li></ol> |
|--|

### *Room Ventilation*

Liquid nitrogen evaporates quickly at room temperature and could replace the air in a room. Use liquid nitrogen in a well-ventilated room ONLY. Important—see the Warning on page ?.

### *Oxygen Absorption*

Liquid nitrogen absorbs oxygen from the air. It is possible for the purity of liquid nitrogen to change as it evaporates from a container. If you suspect that a lot of liquid nitrogen has evaporated, the remaining liquid should be analyzed for oxygen content before being used for any purpose for which a high oxygen content is dangerous.

**!WARNING**

**Potential Asphyxiant**

---

**Liquid nitrogen can cause rapid suffocation without warning.**

**Store and use in an area with adequate ventilation.**

**Do not vent a liquid nitrogen container in a confined space.**

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

The above warning applied to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use. Please refer to the rest of this section for more detailed instructions regarding safety in the use of the Temperature Stage.

---

## *Instrument Symbols*

The following labels are displayed on the  $\mu$ TA™ Sample/Temperature Stage for your protection:

Label	Explanation
	Indicates the presence of high voltage. Turn off the power to the instrument when directed. Follow all safety precautions listed in this manual to avoid injury.

Please heed these labels and take the necessary precautions when dealing with those parts of the instrument. This manual contains cautions and warnings that must be followed for your own safety.

Sample/Temperature Stage

---

## Introducing the Sample/ Temperature Stage

The TA Instruments Sample/Temperature Stage is designed to be used with the TA Instruments  $\mu$ TA™\* (Micro-Thermal Analyzer) 2990, in place of the AFM sample stage originally supplied with the  $\mu$ TA 2990.

Just as with the original sample stage, you can run experiments by affixing the sample to a mount and then placing it on the Sample/Temperature Stage. Then place the  $\mu$ TA 2990 head on the Sample/Temperature Stage and you can begin scanning as described in the  *$\mu$ TA 2990 Operator's Manual*.

You will find that using the Sample/Temperature Stage has the following advantages when performing  $\mu$ TA experiments:

- You can adjust the sample position up and down manually to minimize the need for motor controls on the  $\mu$ TA head. A magnetic plate holds the sample in place, and you can make Z adjustments by turning the knurled wheel to move the sample.
- Rather than having to move the sample to make adjustments in the X and Y directions, you can move the  $\mu$ TA head for finer control of such adjustments.

\* $\mu$ TA™ is a trademark of TA Instruments, Inc.

- The Temperature Stage Controller enables you to image and analyze the sample at other than ambient temperatures: (1) above room temperature or (2) below room temperature with the use of liquid nitrogen.

This manual provides the information necessary to understand, install, operate, maintain, and order parts for the TA Instruments Sample/Temperature Stage.

## *Description*

The Sample/Temperature Stage, shown in Figure 1 on the next page, consists of the following:

- A platform with a magnetic plate on three legs: Two knurled knobs on the sides allow the sample platform to be moved in the X and Y directions. A large knurled wheel at the bottom moves the sample in the Z direction. Two hold-down screws lock the sample platform in place.
- A canister mounted below the sample platform that can be filled with liquid nitrogen for cooling experiments.

### **NOTE:**

- || A canister without temperature control capability (Ambient Sample Stage) is also available.
- A Sample Cover (metal holder and plastic shield) to be mounted on the head during cooling scans: This cover keeps moisture away from the sample when the Temperature Stage is used below room temperature.

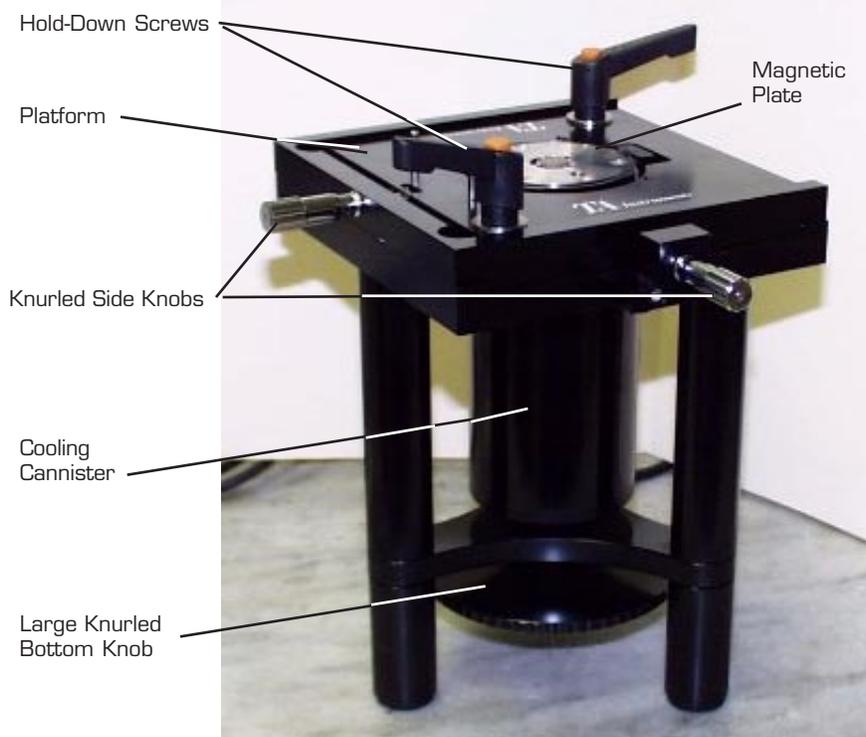
◆ **CAUTION:**

The Sample Cover must be removed if temperatures above 70°C are to be used. The plastic in this part will melt at higher temperatures.

- The Temperature Stage Controller, which is used to control heating and cooling of the sample during scans at other than ambient temperatures.

◆ **CAUTION:**

Do not heat the Temperature Stage above 250°C. Doing so would depolarize the sample mounting magnets.



**Figure 1**  
**The Sample/Temperature Stage**  
**and Controller**

**NOTE:**

The Sample Stage has no electrical connection, connector to a controller, or barrel for temperature control.

*Specifications*

Table 1 contains information about the Sample/Temperature Stage product specifications.

**Table 1**  
**Sample/Temperature**  
**Stage Specifications**

Dimensions Stage	Height: 24 cm (9.5 in.) Width: 18 cm (7 in.) Depth: 22 cm (8.5 in.)
Controller	Height: 8 cm (3.5 in.) Width: 23 cm (9 in.) Depth: 33 cm (13 in.)
Power Requirements	110/220 or 110/240 Vac $\pm$ 10%, de- pending on country
Temperature Range	-75 to 250°C
Heating/Cooling	0 to 90°C/min
<i>Specifications are subject to change</i>	

## Principles of Operation

A DC signal is used to control the temperature of the Stage. A sample, on a conductive mount, is held in place by magnets on the Temperature Stage. A heater mounted under the stage heats the stage to temperatures as high as 250°C. Subambient operation is provided by liquid nitrogen coolant. The heater furnishes heating to overcome the cryogenic temperature of the liquid nitrogen to temperatures as low as -75°C.

The Temperature Stage uses a platinum resistor to sense the temperature. The temperature is displayed by the Stage Controller to 0.1°C. The temperature sensor is located near the sample mount for temperature accuracy.

The Stage Controller has been specifically designed for precise temperature control of the Temperature Stage. The Stage sensor is digitally linearized to give an accurate temperature readout, and the controls and their functions have been carefully chosen for simple and easy operation.

The Temperature Stage is primarily used to maintain the sample at a constant temperature.

◆ **CAUTION:**

Do not attempt to obtain images or perform LTA experiments while the sample is being heated or cooled using the Temperature Stage as you may damage the Z scanner.

During subambient operation, the magnetic plate for the sample is positioned over a metal cold finger, which transmits cold from the canister of liquid nitrogen to the sample. The sample heater,

which surrounds the top of the cold finger, controls the rate and limit of cooling of the sample.

The Sample Stage provides the X, Y, and Z sample movements without temperature adjustment capability.

The Temperature Stage can be used with both Thermal probes (stage temperature range =  $-75$  to  $100^{\circ}\text{C}$ ) and AFM probes (stage temperature range =  $-75$  to  $250^{\circ}\text{C}$ ).

# Installing the Sample/Temperature Stage

The Sample/Temperature Stage is designed to be used with the TA Instruments  $\mu$ TA™ 2990, in place of the AFM sample stage originally supplied with the  $\mu$ TA 2990. This section describes unpacking and installing the Sample/Temperature Stage.

**NOTE:**

It is recommended that you temperature calibrate the Temperature Stage after installation. See page 25 for instruction on temperature calibration.

## *Choosing a Location*

The Sample/Temperature stage is designed to operate safely in the same environment as the  $\mu$ TA 2990, with which it is intended to be used. A vibration-free location, equivalent to that for an analytical balance, is required.

## Power Requirements

The Temperature Stage is either 110/220 or 110/240 Vac ( $\pm 10\%$ ), depending on the country, and requires 15 or 20 amp, 50 to 60 Hz, 1 phase, 1200 W maximum.

◆ **CAUTION:**

If the line power in your laboratory is extremely noisy, an additional power conditioner may be required.

## *Unpacking and Assembly*

The Sample/Temperature Stage was inspected mechanically and electrically before shipment. After unpacking it, inspect the Temperature Stage and Controller for mechanical damage. If it is damaged in transit or fails to operate properly upon receipt, notify the carrier and TA Instruments immediately. Also, check that you have received all items shown on the shipping list. If any item is missing, notify TA Instruments immediately.

**NOTE:**

Small items are sometimes lost in the packing material. Please check through the material carefully before discarding the container.

Follow these instructions to unpack the components of the Sample/Temperature Stage. As you remove each piece, place it in a safe location (*i.e.*, an area where it will not be knocked to the floor, etc.).

1. Locate and unpack the Sample/Temperature Stage. The rubber feet are packed separately in the same container along with the sample cover.
2. Affix a rubber foot into the bottom of each leg of the Stage by (a) removing the release sheet from the foot to expose the adhesive and (b) pressing the foot onto the leg with the adhesive side against the bottom of the leg. Place the Stage on the work table within reach of the  $\mu$ TA 2990.

3. Remove the Sample Cover (used during cooling scans) and funnel (for filling the canister with liquid nitrogen) from the packaging.
4. Locate and unpack the Stage Controller and its power cable and connect it as follows:
  - a. Plug the cable into the back of the controller, as shown in Figure 2.
  - b. Plug the power cord of the controller to a main outlet).
5. Connect the cable from the Stage to the back of the controller, as shown in Figure 2.



**High voltage is present at the PC board and connectors on the Sample/Temperature Stage and Controller. *Always* take extreme care to avoid direct contact while the instrument hardware is powered on. Always turn off the power to the equipment before attempting to remove any panels or PC boards, and before touching any connectors by hand or with electrically conductive tools.**



**Figure 2**  
**Rear Panel of Stage Controller**

## Installing the Sample Cover

You should install the sample cover on the Temperature Stage before you scan using the cooling function. Otherwise, the sample area would frost up during cooling, interfering with scanning results.

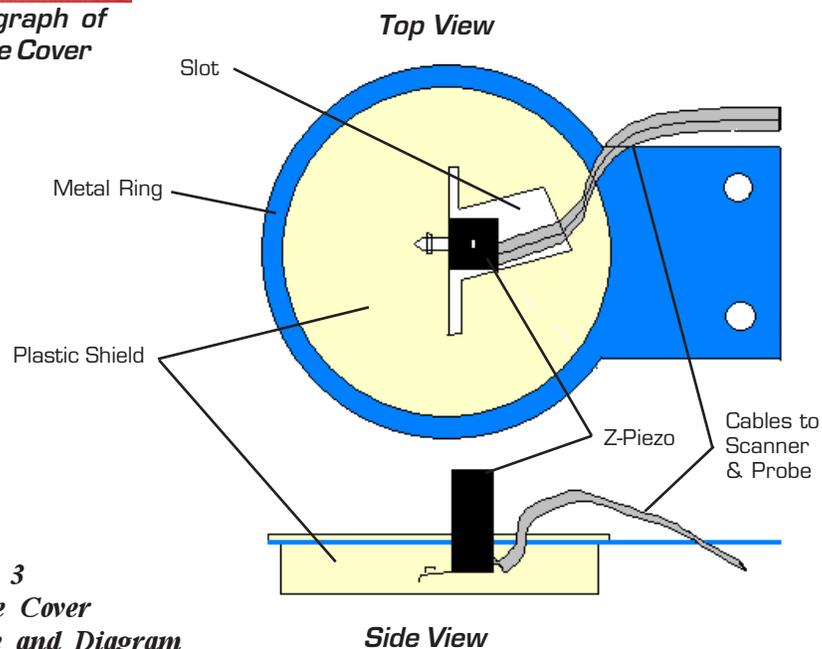
Use the following procedure to install the sample cover (see Figure 3).

◆ **CAUTION:**

The sample cover must be removed if temperatures above 70°C are to be used. The plastic in this part will melt at higher temperatures.



**Photograph of Sample Cover**



**Figure 3**  
**Sample Cover**  
**Picture and Diagram**

1. Remove the probe first.
2. Assemble the sample cover, if necessary, by putting the plastic shield into the ring.
3. Mount the sample cover onto the Temperature Stage so that the Z-scanner fits through the square opening at the top of the plastic shield with the open side of the shield facing downward (*i.e.*, toward the sample). See Figure 3.
4. Remove the dress shield covering the Z-scanner connections. Mount the sample cover in the position previously occupied by the dress shield. Ensure that:
  - The long, thin slot in the plastic shield somewhat parallels the X scanning angle of the Z-scanner, with the curb being close to the scanner.

*and*

  - The wider hole perpendicular to the slot is located at the back of the scanner and pointing away from the probe tip (see Figure 3).
5. Pull all the cables through the wider hole so that later, the bottom of the plastic piece will close the gap to the stage.
6. Replace the probe tip. To facilitate laser adjustment, make sure that the plastic shield is not tilted.

**NOTE:**

Use of the sample cover results in a loss of some laser power, so the reading on the sum signal will be decreased by 25% to 50%.

## Filling the Cooling Canister

Follow these steps to fill or refill the Temperature Stage cooling canister with liquid nitrogen, using the funnel supplied with the Stage (see Figure 4 on the next page). The canister holds enough liquid nitrogen for about 30 minutes of cooling, depending on the target temperatures used.



**Liquid nitrogen evaporates rapidly at room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.**

1. Press the **Start/Finish** button on the Stage Controller.
2. Turn the **Limit** knob on the controller counterclockwise until the limit value, or target temperature, is “25.” The Sample/Temperature Stage will now hold at 25°C while you fill the canister.
3. Remove the  $\mu$ TA head from the stage.
4. Place the funnel on the stage with its tip in the hole.
5. Align the tip of the funnel with the hole in the top of the cooling canister. Then use the lower knurled knob to bring the canister up around the bottom of the funnel so its tip is inside the canister.
6. Fill the canister with liquid nitrogen.

7. Remove the funnel, and replace the  $\mu$ TA head.



***Figure 4***  
***Filling the Canister with***  
***Liquid Nitrogen***

---

## Operating the Sample/ Temperature Stage

You can use the Temperature Stage in either of two ways:

- As a simple sample stage for scanning at ambient temperature, like the  $\mu$ TA 2990's sample platform: The Stage permits X and Y movement of the mounted sample to locate scanning areas. It also allows you to raise and lower the sample platform under the probe, to limit the use of the motor for alignment.
- To heat or cool the sample to a desired temperature for imaging and  $\mu$ TA.

### *Basic Procedure*

The basic procedure for using the Sample/  
Temperature Stage and Stage Controller is as follows:

1. Prepare the sample as directed in the  *$\mu$ TA 2990 Operator's Manual* for the type of sample.
2. Open the  $\mu$ TALab Software on your computer, and prepare for scanning or Localized Thermal Analysis (see the  *$\mu$ TA 2990 Operator's Manual*).
3. Using tweezers, mount the sample on the magnets on the sample area of the Stage.

4. Move the sample in the X and Y directions using the knurled knobs on either side of the stage. Once you have achieved the desired position, you can lock the mechanical stage in place using the locking handles.
5. Use the Z-height knob to move the sample up or down below the stage surface.

**NOTE:**

The sample moves slightly upward during lock down. This could damage the probe if it is too close to the sample surface.

6. If you will be cooling the sample for scanning, mount the sample cover on the probe. Also, fill the canister with liquid nitrogen if needed (see page 13).

**◆ CAUTION:**

**DO NOT** use the sample cover when scanning at temperatures above 70°C. The plastic in this part will melt at higher temperatures.

7. Use the Z-height knurled wheel on the Stage to move the sample up toward the probe.
8. Using the Stage Controller, adjust the temperature of the stage to the desired value (see next page).

**◆ CAUTION:**

**Do NOT** heat the Temperature Stage above 100°C when using the standard Wollaston thermal probe. An epoxy cement is used in the probe that will soften above 100°C.

9. Press the **Start** button on the Stage Controller panel to start the heating/cooling program.
10. Click on the **Start Scan** button, , in the  $\mu$ TALab Software to start scanning.

## Using the Stage Controller

Press the Off/On switch on the rear panel to turn on the power to the Temperature Stage Controller (see Figure 5).

As shown in Figure 5, the front panel of the Stage Controller contains a display screen, a **Limit** knob, six buttons, and three indicator lights.



**Figure 5**  
*Temperature Stage  
Controller Front Panel*

When the Stage Controller is powered on, the display screen shows “TA Instruments,” and the **Power** LED (green) lights up. The display then changes to show three readings (left to right): rate, current temperature, and target temperature. The values displayed depend on the ambient temperature and previously selected values.

The **Load** LED (red) flashes when the **Start/Finish** button is pressed to start a heating or cooling program. The **Cool** LED flashes whenever a cooling program or ramp is being executed; it goes off when a target temperature is reached.

## Setting the Rate

Three buttons on the Stage Controller, as their names imply, are used to choose the heating or cooling rate: Rate/10, Rate x 10, and Rate Step. Three ranges are available for the rate, as shown in Table 2; most work will be performed between 10 and 20°C/min.

**Table 2 Ranges for Heating/Cooling Rate**

0.1 through 0.9°C/minute, in increments of 0.1
1 through 9°C/minute, in increments of 1
10 through 90°C/minute, in increments of 10

Use the **Rate Step** button to change the heating or cooling rate. A single press of this button raises the displayed value by one unit, either 0.1, 1.0, or 10, depending on the value first displayed when the unit is powered on. For example:

- If the first displayed value is “10,” the first press of the **Rate Step** button will change it to “20,” a second press to “30,” and so on to “90,” after which another press of the button returns the value to “10.”
- If the first displayed value is “1.0,” the first press of the **Rate Step** button will change it to “2.0,” a second press to “3.0,” and so on to “9.0,” after which another press of the button returns the value to “1.0.”

- If the first displayed value is “0.1,” the first press of the **Rate Step** button will change it to “0.2,” a second press to “0.3,” and so on to “0.9,” after which another press of the button returns the value to “0.1.”

To switch to another range for the **Rate** value, use the **Rate/10** or **Ratex10** button. For example:

- If the displayed value is “10,” but you wish to use a value between 1 and 9, press the **Rate/10** button once; the value changes to “1.” Then use the **Rate Step** button to select the value you want in that range.
- If the value displayed is “10” but you wish to use a value between 0.1 and 0.9, press the **Rate/10** button twice; the value changes to “0.1.” Then use the **Rate Step** button to select the value you want in that range.
- If the value displayed is “1” but you wish to use a value between 10 and 90, press the **Ratex10** button once; the value changes to “10.” Then use the **Rate Step** button to select the value you want in that range.
- If the value displayed is “0.1” but you wish to use a value between 10 and 90, press the **Ratex10** button twice; the value changes to “10.” Then use the **Rate Step** button to select the value you want in that range.

**NOTE:**

If an asterisk begins to flash in the display after you have set a cooling rate, you have selected a rate that is greater than the natural cooling rate of the Stage.

Page 24 contains a sample procedure with instructions on how to program the Stage Controller.

## Selecting the Target Temperature

Select the target temperature for your heating or cooling program by turning the **Limit** knob on the front panel of the Stage Controller (see Figure 6). Turning the knob clockwise raises the displayed value; turning it counterclockwise lowers the displayed value.

The values available on the Stage Controller for target temperature range from  $-200$  to  $+600^{\circ}\text{C}$ , in whole numbers. However, the lower and upper limits for the actual Temperature Stage are  $-70$  and  $250^{\circ}\text{C}$ .

◆ **CAUTION:**

Do not heat the Temperature Stage above  $250^{\circ}\text{C}$ . Doing so would depolarize the sample mounting magnets.

◆ **CAUTION:**

The Sample Cover must be removed if temperatures above  $70^{\circ}\text{C}$  are to be used. The plastic in this part will melt at higher temperatures.

You can also use the **Limit** knob to cancel a heating or cooling ramp while the Stage Controller is running, by turning the knob to a limit higher or lower than the present temperature of the stage.

## Using the Start/Finish Button

As its name implies, the **Start/Finish** button starts or stops the heating or cooling program whenever it is pressed. When you have mounted a sample and entered the parameters for heating, cooling, or isothermal scanning, press the **Start/Finish** button. The **Load** LED begins to flash to indicate that the Stage Controller is working. The Stage Controller either heats or cools at the rate until the limit is reached.

During a heating or cooling program, pressing the **Start/Finish** button halts the program.

## Using the Hold Button

When the **Hold** button is pressed, the Stage Controller keeps the temperature of the Stage at the current value (Hold mode). This button functions only if the Sample/Temperature Stage is actually heating or cooling. When the **Hold** button is pressed, the screen alternately displays the word “Hold” and the current temperature.

You can also use Hold mode to change the heating/cooling rate and target temperature. Press the **Hold** button; then:

- Use the **Rate** buttons and/or the **Limit** knob to change the values as needed.
- Press the **Heat/Cool** button to start the temperature change in the opposite direction (see next page).

## Using the Heat/Cool Button

The **Heat/Cool** button functions only when a heating or cooling program is running or the Stage Controller is in Hold mode (because the **Hold** button has been pressed). Pressing the **Heat/Cool** button after the **Hold** button has been pressed cancels the Hold mode. Whether the Stage Controller is in hold mode or is actively heating or cooling, pressing the **Heat/Cool** button stops the current program and sends the temperature in the opposite direction.

- If the Stage Controller is heating to a limit (target temperature), pressing the **Heat/Cool** button starts it cooling. The word “Cool” is displayed where the target temperature value was. Once the temperature reaches the target temperature for the Temperature Stage, the Stage Controller will stop cooling.
- If the Stage Controller is cooling to a limit (target temperature), pressing the **Heat/Cool** button starts it heating. The word “Heat” is displayed where the target temperature value was. Once the temperature reaches the target temperature for the Temperature Stage, the Stage Controller will stop heating.

◆ **CAUTION:**

Do not allow the Temperature Stage to be heated above 250°C. Doing so would depolarize the sample mounting magnets.

You can override the new ramp direction set by the **Heat/Cool** button by manually changing the limit using the **Limit** knob.

## Sample Procedure

The following procedure describes how to program the Stage Controller to cool a mounted sample at a rate of 10°C/min to -30°C.

1. Turn on the power to the Stage Controller.
2. The **Rate** value (value on left of display) first displayed is “2” (°C/min), and the limit value (value on far right of display) is “80” (°C).
3. Press the **Ratex10** button once. The display now reads “20.”
4. Press the **Rate Step** button 8 times, until the rate value is “10.”
5. Press the **Start/Finish** button on the Stage Controller.
6. Turn the **Limit** knob counterclockwise until the limit value, or target temperature, is “25.” The Stage will now hold the temperature to 25°C while you fill the canister with liquid nitrogen.
7. Fill the canister with liquid nitrogen (see page 12).
8. Turn the **Limit** knob counterclockwise until the limit value or target temperature is “-30.”
9. Press the **Start/Finish** button to begin cooling your sample to -30°C at a rate of 10°C/min.

## Performing Temperature Calibration

It is recommended that temperature calibration be performed upon installation of the Temperature Stage. The procedure for obtaining and entering new values for the positive calibration points (for points greater than 0°C) is described here; you can also obtain new calibration values for the zero point and for negative points (less than 0°C).

### *Removing the Old Calibration Factors*

Before a new set of calibration values can be determined on the Temperature Stage, the existing calibration factors must be removed. This step ensures that the Stage Controller will read the sensor resistance directly and convert without using any calibration factors. Removing the old calibration values resets the existing measured and actual values to be the same, and the zero point to be reset to zero.

Remove the old values for the positive calibration factors as follows:

1. Turn the power for the Stage Controller off and then back on.
2. As soon as the display lights up, press the **Heat/Cool** button. The display will then read "Sample Calibrate."

3. Press the **Rate Step** button. The display will then read “Step=Reset Calib.” The Temperature Stage and Stage Controller are now reset, so new calibration values may be obtained.

## *Obtaining New Positive Calibration Factors*

The example given here is for calibrating for values above 0°C; for calibration below 0°C, use a material with a known melting temperature below 0°C and enter Values 1 and 2 when prompted.

For this procedure, you will need the following items, which can be found in your  $\mu$ TA™ 2990 accessory kit:

- A few milligrams of a temperature reference material (*i.e.*, a material with a known melting temperature), such as anisic acid or biphenyl.
- An open (*i.e.*, without a lid) DSC sample pan.

### **NOTE:**

|| Samples used for temperature calibration are mounted at room temperature.

1. Place the temperature reference material in the open DSC sample pan, and put the pan on the Temperature Stage in place of the sample mount.
2. Bring the Temperature Stage to a temperature 5°C below the anticipated calibration temperature.

3. Set up a temperature program on the Stage Controller to heat at a **Rate** of 1°C/min to 5°C above the reference material's known melting temperature.
4. Press **Start** on the Stage Controller panel to begin the calibration, and watch the sample as it is heated.
5. When the last of the sample melts, check and record the temperature shown on the Stage Controller display.
6. Press the **Start/Finish** button to halt the program, and remove the sample.

You can now enter the new calibration factors.

### *Entering the New Calibration Factors*

1. Turn the power for the Stage Controller off and then back on.
2. As soon as the display lights up, press the **Heat/Cool** button. The display will first read “Sample Calibrate,” and then will change to “Step=Reset Calib.”
3. Press the **Heat/Cool** button again. The display will read “Value 1 ?.”
4. Turn the **Limit** knob until the *actual* positive calibration value—that is, the actual melting temperature of the sample reference material—is displayed. For example,

Value 1: [biphenyl] 69°C

5. Press the **Hold** button. The display will change to read “Value 2 ?.”
6. Turn the **Limit** knob again until the measured positive value—that is, the temperature you recorded from the display when the sample material had completely melted.

For example:

Value 2: 70°C

The Stage Controller has now been calibrated, and the display will now show the measured temperature modified by the calibration factor.

# Index

---

## Symbols

$\mu$ TA 2990 1-1

$\mu$ TALab Software 1-15

## A

adjustments

    X and Y direction 1-1

    Z direction 1-1

assembly 1-8

## B

basic procedure 1-15

## C

canister 1-2

    filling with liquid nitrogen 1-13

coolant 1-5

cooling 1-5

cooling canister. *See* canister

## D

description 1-1

## E

electrical

    WARNING 1-9

## H

Heat/Cool button 1-23

high voltage ix

Hold button 1-22, 1-23

hotlines to TA Instruments v

## I

installation 1-7 to 1-10

    sample cover 1-11

Instrument Symbols xiii

L

Licensing Agreement ii

Limit knob 1-17

liquid nitrogen 1-5, 1-13

Load LED 1-17

location 1-7

M

magnetic plate 1-1, 1-2

N

Nitrogen  
liquid  
handling safety x

notes, cautions, and warnings viii

O

operation 1-15 to 1-24  
Sample/Temperature Stage 1-15

P

platinum resistor 1-5

Power LED 1-17

power requirements of instru-  
ment 1-7

principles of operation 1-5

R

rate  
setting with the Stage Control-  
ler 1-18

Rate Step button 1-18

Rate/10 button 1-19

Ratex10 button 1-19

reference material 1-26

S

Safety  
liquid nitrogen x  
safety ix to x

---

sample	T
moving up or down 1-16	
Sample Cover 1-2	TA Instruments
	addresses and phone numbers v
sample cover	target temperature 1-20
installing 1-11	temperature calibration 1-25 to 1-28
mounting 1-12	entering new positive calibration
sample heater 1-5	factors 1-27
sample pan 1-26	obtaining new positive calibration
sample platform 1-2	factors 1-26
	removing old calibration factor 1-
	25
sample procedure 1-24	
Sample/Temperature Stage	U
description 1-1	
sensor resistance 1-25	unpacking 1-8
specifications 1-4	
Stage Controller 1-2, 1-5	V
setting the rate 1-18	
using 1-17	voltage
Stage sensor 1-5	WARNING 1-9
Start/Finish button 1-21	Z
subambient operation 1-5	Z adjustments 1-1

Sample/Temperature Stage

---