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Thermal Analysis & Rheology A Subsidiary of Waters Corporation



Operator's Manual

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Notes, Cautions and Warnings

NOTE:	to emphasize important and critical instructions.
♦ CAUTION:	A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.
! WARNING	A WARNING indicates a procedure that may be hazardous to the operator or to the envi-

EMC Specifications

In order to comply with the requirements of Council Directive 89/336/EEC (EMC Directive), the GCA has been tested and found to comply with the following standards on Electromagnetic Compatibility:

 Emissions: EN 55011: 1991, CISPR 11:1990 Group 1 Class B (30-1000 MHz) Radiated

EN 55011: 1991, CISPR 11:1990 Group 1 Class B (0.15-30 MHz) Conducted

- Immunity: EN 50082-1: 1992, Electromagnetic Compatibility-Generic immunity standard Part 1. Residential, commercial, and light industry.
- IEC 801-2: 1991. Electrostatic Discharge.
- IEC 801-3: 1984. Radiated RF Immunity.
- IEC 801-4: 1988. Electric Transients.

Safety

Read this section and understand the Safety precautions before proceeding with installing or using the GCA.

In order to comply with the requirements of Council Directive 73/23/EEC (Low Voltage Directive), amended by 93/68/EEC, the GCA has been evaluated and tested, and found to comply with the following standards on safety:

- IEC 1010-1/1990, A1/1992, and A2/1995
- IEC 1010-2-010/1992, A1/1996
- EN 61010-1/1992
- EN 61010-2-010/1994
- UL 3101-1, First Edition.

Instrument Symbols

The following label is displayed on the Gas Cooling Accessory for your protection:

Symbol	Explanation
	Indicates the presence of a hazard related to the area or part indicated.

Please heed these labels and take the necessary precautions when dealing with those parts of the accessory. The *Gas Cooling Accessory Operator's Manual* contains cautions and warnings that <u>must</u> be followed for your own safety. Refer to the information in this section for specific information related to safety.



Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent GCA container in confined spaces.

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

The warning above applies 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 GCA.

Safety

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°C) it will burn 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 and wear gloves that are easily removed. Wear high-topped shoes with pant legs *outside* the tops for extra protection.
- 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.
- 3. Use only approved low temperature containers.
- 4. Make sure liquid nitrogen containers are vented to prevent pressure buildup.

If Burned by Liquid Nitrogen

- 1. Flood the area (skin or eyes) IMMEDI-ATELY with large quantities of cool water, then apply cold compresses.
- 2. See a doctor IMMEDIATELY if the skin is blistered or if the liquid nitrogen came in contact with your eyes.

Room Ventilation

Liquid Nitrogen evaporates quickly at room temperature and could replace the air in a room. Only use liquid nitrogen in a well-ventilated room. Important—see the Warning on page *ix*.

Oxygen Absorption

Liquid Nitrogen will absorb oxygen from the air. It is possible for the purity of liquid nitrogen to change as it evaporates from a container. If you suspect a lot of liquid nitrogen has evaporated the remaining liquid should be analyzed for oxygen content before using if for any purpose where high oxygen content is dangerous.

Safety

Pressure Buildup

Liquid Nitrogen should not be stored in a sealed container, as tremendous pressure could result and an explosion is possible.

The GCA is designed to always be vented to the room when not supplying nitrogen gas to the test instrument. The pressure build up in the GCA, when it is supplying nitrogen gas to the instrument, is limited by the controller. Pressure relief valves are also designed into the system.

If the feed line pressure relief valve is venting, either the bulk storage tank pressure is too high, or the bulk storage tank valve has been closed, trapping liquid nitrogen in the feed tube. Verify that gas is flowing through the vent and coolant feed valve before continuing normal operation by running the following method:

- 1 Jump to -50°C
- 2 Isothermal for 1 min.
- While the method is running vapor should be coming from the DMA furnace assembly. If no vapor is apparent, stop the method and check the coolant transfer tube for blockage. If no blockage is found, call TA Instruments for service. If a blockage is found, clear it and run the method again.
- When the method has completed, you should hear gas venting from the GCA. If you do not hear the gas venting, call TA Instruments for service.

Safety

♦CAUTION:	Always make sure the GCA system is installed correctly. Make sure the fill line from the bulk storage tank is hooked up properly.	
♦ CAUTION:	Never allow liquid nitrogen to be trapped in the fill line from the bulk storage tank to the GCA.	
	The sequence for opening and closing valves is important to prevent trapping liquid nitrogen in the fill tube. When the bulk storage feed tube is connected for autofilling, it is important that you do not ever close the manual valve on the bulk storage tank, unless the bulk storage tank is empty, or at least 15 minutes has elapsed since the solenoid valve at the GCA has closed. (The solenoid valve closes at the end of Autofill.) This time allows the liquid nitrogen to vaporize before sealing the area between the solenoid valve and the valve on the bulk storage tank.	
♦CAUTION:	Never remove the GCA Autofill line at the bulk storage tank without closing the bulk storage tank valve first.	
	When connecting and removing the GCA Autofill line remember to wear goggles and gloves.	
I warning	Do not use high pressure bulk tanks [greater than 25 psi (175 kPa)]. The GCA is designed for lower pressure bulk tanks. Using high pressure tanks will cause the GCA to work improperly and raise the potential for injury.	

Water Condensation

The GCA surfaces get cold during use of the GCA for both filling and supplying cold nitrogen to the instrument. The cold surfaces cause condensation and, in some cases, frost to build up. This condensation may drip to the floor. Provisions to keep the floor dry should be made. If any moisture does drip to the floor, be sure to clean it up promptly to prevent a slipping hazard.

Electrical Safety

	High voltages (120 Vac) are present in this instrument, only qualified service personnel should remove covers and make repairs.
WARNING	The power at the instrument <i>must</i> be turned off, and the interface cable and power cord must be removed before any service or repair work is started.
W ARNING	Hazardous voltage is present inside the GCA. Do not remove the clamp securing the cap to the dewar. There are no user-serviceable parts inside the GCA. Call TA Instruments for service.

Lifting the Instrument

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



Roll the GCA on its wheels to move it, whenever possible. If you must lift it, use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

Chapter 1: Introducing the GCA

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Introducing the GCA

IntroductionThe GCA (Gas Cooling Accessory) is a cooling
accessory for use with the TA Instruments
Dynamic Mechanical Analyzer (DMA) 2980.The GCA has been designed for automatic
refilling from a low pressure [25 psi (175 kPa)
maximum] bulk storage tank that can be located
within 6 feet (1.8 meters) of the GCA. The
GCA is also capable of being filled manually by
disconnecting it from the instrument and moving
it to a bulk storage source.NOTE:Before proceeding, be sure you understand and
follow the safety precautions in the prefix of this
manual.

Specifications

The specifications in Tables 1.1, and 1.2 apply to the Gas Cooling Accessory.

Table 1.1GCA TechnicalSpecifications

GCA Liquid Nitrogen Capacity	50 Liters
Size	107 cm (42") high 46 cm (18") in diameter
Power requirements	120 volts at 8 amps, 50-60 Hz.
Weight	empty: 100 lbs (45.5 kg) full: 190 lbs (86.4 kg)
Cooling Capacity	-145°C
Pressure Relief	10 psi (70 kPa) relief valve on tank. 50 psi (345 kPa) on fill line.
_	(table continued)

Specifications

Liquid Nitrogen Tubes	Transfer: 6 ft (1.8 m) insulated from GCA to instrument.
	Feed: 6 ft (1.8 m) insulat- ed with shutoff solenoid from bulk storage to GCA.
GCA Fill Modes	Automatic: Bulk storage within 6 ft (1.8 m) of GCA.
	Manual: Remote filling at bulk storage location.
Bulk Storage Tank	Use low pressure supply tank only. Recommended filling pressure is 20 to 25 psi (140 to 170 kPa).
	Liquid Nitrogen Tubes GCA Fill Modes Bulk Storage Tank

NOTE:

The GCA vents to the atmosphere, if no filling or testing is currently in progress.

Theory of Operation

The GCA uses up to eight selectively switched 100-watt heaters to vaporize the liquid nitrogen and obtain required pressures of up to 9 psi (62 kPa). The pressurized gas is forced out of the tank and into the DMA 2980 furnace assembly. The number of heaters that are turned on depend on the cooling rate desired.

The operation of the GCA is very simple. When cooling gas is needed by the instrument, the following events occur (refer to Figure 1.1 for an illustration of the numbered parts):

- 1. Instrument requests cooling gas.
 - 1. Heater in the GCA is turned on.
 - 2. Feed solenoid valve #1 opens.
 - 3. Vent valve #2 is closed.
- 2. Instrument no longer requests coolant.
 - 1. Heater in the GCA is turned off.
 - 2. Feed Solenoid valve #1 closes.
 - 3. Vent valve #2 is opened.



Figure 1.1 GCA Schematic Operation

Description of Components

The following illustration shows the major parts of the TA Instruments GCA. Refer to Table 1.2 for a descriptions of these parts.



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Item	Description
50 Liter dewar	The thermally-insulated storage vessel for liquid nitrogen.
Bulk Storage Feed Tube	Allows the automatic and manual filling of the 50 liter GCA dewar from a bulk storage source.
Coolant Transfer Tube	Supplies gaseous nitrogen from the GCA to the instrument.
Feed Solenoid Valve	An automatic solenoid valve that opens to supply gaseous nitrogen to the instrument.
Interface Cable	Provides the control signals from the instru- ment to the GCA.
GCAFill Valve	<i>(not shown)</i> An auto- matic solenoid valve that is used to refill the GCA automatically.

Table 1.2GCA Components

Introducing the GCA

Top Section of GCA

The top section of the GCA contains several items important to the operation of the accessory and the instrument. Figure 1.3 illustrates the parts of the top left side of the GCA. Refer to Table 1.3 for a description of the individual parts.





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Table 1.3 Parts of the Top Laft Section of the CCA		
Left Section of the UCA	Item	Description
	Instrument Connection	Connects the GCA with the instrument to commu- nicate information such as GCA tank pressure, heater regulation, etc. between the GCA and the instrument.
	Accessory Connection	Connects the GCA to accessories such as the Gas Switching Accessory.
	Load	Used to place the GCA in load mode. See "Updat- ing the Software" for instructions.
	Ready Lamp	Glows when the GCA has finished its confidence test and is ready for operation with the instrument. If this lamp blinks, it signals a fatal error during the confidence test—call TA Instruments for service.
	Reset Button	Press to reset the GCA and run the confidence test again.

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Several other parts of importance to the operation of the GCA are contained on the back of the top section as seen in the figure below. See Table 1.4 for a description of these parts.



Figure 1.4 Back of the Top Section of the GCA

Table 1.4Parts on the Backof the Top Section

ItemDescriptionBulk Storage AutoFill ConnectionAllows the GCA to be refilled automatically by connecting a bulk storage tank.ON/OFF SwitchTurns the GCA power on. This switch must be on for the GCA to supply coolant to the instrument and to refill the GCA automati- cally from the bulk liquid nitrogen storage container.Power CablePlugs into a source of electrical power to provide power for the operation of the GCA and its heaters.FuseAn 8A, 125/250V normal blow fuse is located in the top section of the GCA. If this fuses blows, you will get no response from the unit when you attempt		1
Bulk Storage AutoFill ConnectionAllows the GCA to be refilled automatically by connecting a bulk storage tank.ON/OFF SwitchTurns the GCA power on. This switch must be on for the GCA to supply coolant to the instrument and to refill the GCA automati- cally from the bulk liquid nitrogen storage container.Power CablePlugs into a source of electrical power to provide power for the operation of the GCA and its heaters.FuseAn 8A, 125/250V normal blow fuse is located in the top section of the GCA. If this fuses blows, you will get no response from the unit when you attempt	Item	Description
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Fuse An 8A, 125/250V normal blow fuse is located in the top section of the GCA. If this fuses blows, you will get no response from the unit when you attempt	Power Cable	Plugs into a source of electrical power to provide power for the operation of the GCA and its heaters.
to turn it on.	Fuse	An 8A, 125/250V normal blow fuse is located in the top section of the GCA. If this fuses blows, you will get no response from the unit when you attempt to turn it on.

Introducing the GCA

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Installing the GCA

Unpacking and Inspecting

By the time you are reading this manual, you have already done a certain amount of unpacking. Continue to unpack and inspect the contents of the GCA shipping box. Retain the shipping container and packing materials until the unit has been successfully installed and verified to be functioning correctly.

If the GCA received rough handling in shipment and signs of damage are apparent, contact the carrier immediately for advice on how to make a claim. Please call TA Instruments to advise us of the problem. DO NOT use or install the instrument until an authorized representative of TA Instruments has repaired it.

Contact your TA Instruments representative if parts are missing.

Before Installation

There are a few items to check before you begin installing the GCA to the instrument. Please check the following:



Read the safety precautions for handling cryogenic materials (located in the prefix of this manual) before installing the GCA. Wear goggles or a face shield and gloves large enough to be removed easily whenever you handle liquid nitrogen.

- Check the clamp holding the GCA top section to the dewar. The clamp is located below the sheet metal covers. The clamp must be tight for proper operation of the equipment.
- Connecting the GCA to an instrument requires that a specific interface cable be used. Make sure the proper cable is being used for this installation.

Choosing a Location

Because of the sensitivity of experiments using the GCA, it is important to choose a location using the following guidelines:

- *In* . . . a temperature-controlled area.
 - . . . a clean environment.
 - ... an area with ample working and ventilation space around the instrument. (Refer to the technical specifications in Chapter 1 for the instrument's dimensions.)
- Near . . . a power outlet (120 Vac, 50 or 60 Hz, 10 amps). A step up/down line transformer may be required if the unit is operated from a higher or lower line voltage.
 - ... your TA Instruments DMA 2980.

Away

from dusty environme	nts.
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.

- . . . exposure to direct sunlight.
- . . . direct air drafts (fans, room air ducts).
- . . . poorly ventilated areas.

After you have decided on the location for your instrument and GCA, refer to the next several sections to unpack and install the GCA.

Help Lines

United States of America

For Technical Assistance	e (302)427-4070
To Order Instruments and Supplies	
For Service Inquiries	
Sales	
m t T i i i	

TA Instruments has a worldwide network of representatives, please refer to Appendix B for a list of subsidiaries. To obtain more detailed information about how to contact your local representative, call the Sales number above or send a request by Fax to International Sales at (302)427-4186.

Installing the GCA

Installing the GCA primarily consists of attaching the cables and lines to the instrument and the bulk liquid nitrogen source. Use the following basic steps to install the GCA.

- 1. Position the GCA within 6 feet (1.8 meters) of the instrument.
- Attach the coolant transfer tube to the GCA at the fitting labeled N₂ Gas to Instrument/ LN₂ Manual Fill.
- 3. Remove the air cool line (if present) from the 90° elbow fitting on the DMA, and attach the coolant transfer tube (see the figure below). Make sure the fittings are tight.



Figure 2.1 Attaching the Coolant Transfer Tube to the DMA

4. Make sure the power switch on the GCA is in the off position.

♦ CAUTION: The instrument power switch should be off before connections are made.

 Connect the interface cable to left side of the GCA at the connection marked IN-STRUMENT as shown in the figure below.



GCA Top Section

6. Connect the other end of the interface cable to the rear of the instrument at the connection marked **Serial**, as shown in the figure on the next page.



- 9. Arrange the low pressure bulk storage source physically close enough, within 6 ft (1.8 m), to the GCA so that the autofill tube can be easily connected between the source and the GCA. Likewise the GCA and the instrument need to be in close proximity to allow connection of the control cable and the 6-ft (1.80-m) coolant transfer tube.
- 10. Attach the bulk storage feed tube to the GCA. Use a wrench to tighten the bulk storage autofill connector. See the figure below.





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11. Attach the other end of the bulk storage feed tube assembly to the liquid feed connector on the bulk storage container.



Figure 2.5 GCA Attached to DMA 2980

TA INSTRUMENTS GAS COOLING ACCESSORY

Installing the GCA

Installing the Drain Valve

Ice and frost are created during normal use of the Gas Cooling Accessory. The GCA catch trough is designed to prevent water from dripping onto the floor creating a potential hazard when the ice and frost melt.

The drain valve may be needed to occasionally empty water from the catch trough. To install the condensate drain valve, use a 5/8-inch wrench on the swage nut, screw the elbow into the fitting until it is hand tight with the valve pointing down (see the figure below).



Figure 2.6 Drain Valve Installation

TA INSTRUMENTS GAS COOLING ACCESSORY

Empty the GCA catch trough periodically by opening the valve and draining the water into a suitable container, or a hose can be connected to the valve and routed to a floor drain or large container.

♦ CAUTION: During manual filling operations, do not over fill the GCA tank causing liquid nitrogen to spill into the catch trough.

The software used to run the GCA has already been loaded at TA Instruments. However, you may need to update that software with a new version. Follow the instructions in the next section to update the GCA program.

After the GCA has been completely installed, and the software updated, if necessary, you can turn to the next chapter for the instructions needed to fill the accessory with liquid nitrogen. Installing the GCA

Updating the Software

If you need to update the software with a new version, follow these instructions to connect the GCA to the controller and to run the TA Instruments Accessory Loader program: NOTE: The windows appearing in this section reflect the Thermal Solutions/Advantage software. If you are using the OS/2 controller software, the windows will appear slightly different but the procedure is still basically the same. 1. Disconnect the RS-232 interface cable from the DMA (see pages 2-8 and 2-9 for location). 2. Connect the RS-232 interface cable from your GCA to your controller. (You may need to disconnect both ends of the RS-232 cable, then reverse the ends so that the proper gender connectors match.) NOTE: You can connect the RS-232 cable from the controller to either the INSTRUMENT or ACCES-SORY connection on the GCA. 3. Start the TA Instruments Accessory Loader program from the Start/Programs menu. The window shown in the figure on the next page is displayed. 4. Select the **Start** button to begin loading the program. The program will check to make sure a TA Instruments accessory is connected to a communications (COM) port on the controller (see pages 2-8 to 2-9 to

connect the GCA to the controller).

The button will change to read **"Pause,**" to allow you to halt the loading process, if desired.

	a∓ TA Insl	truments Access	ory Loader			? ×
Status Message Area	Downloa	ad Status	Ready for	Operation		
Percent Complete Area	0	20	40 1	60 I	80 , I	100
Start\Pause\ Resume\Exit Button		<u>S</u> ettings	<u>S</u> t. <u>A</u> b	art	<u>H</u> elp	

Figure 2.7 Accessory Loader Window

Refer to Table 2.1, on the next page, for a description of the parts of the Main window.

Installing the GCA

Table 2.1Parts of theMain Window

Part	Description
Status Message Area	As the program is loading, this area will display text describing the action in progress.
Percent Complete Area	A status bar is filled in from left to right as the program loads, providing a visual method to track the percent completion of the procedure.
Start/ Pause/ Resume/ Exit Button	This main control button changes depending on the current point in the loading process. "Begin" is used to start the loading process. "Suspend" is used to halt the program and place it on hold. "Resume" is used to restart the program after it was suspended. "Exit" is used, after the program has completely loaded the software, to end the program.

If no GCA is found, you can change the program settings, if needed, by following the instructions found in the section entitled "Changing the Settings" on page 2-20.

If the GCA is located successfully, the following message is displayed.



Figure 2.8 Load Button Message



3. Locate the recessed Load button on the side of the GCA, shown in the figure below.

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4.	Using a blunt-end tool, such as a pen tip,
	depress the Load button. The Ready lamp
	should go off to indicate that you have
	successfully placed the accessory into the
	"load" state.

5. Click the **Resume** button on the **Accessory Loader** window.

NOTE: You can halt and cancel the download process by selecting the **Cancel** button.

The window shown below is displayed.

Accessory D	ownload File		? ×
Look jn:	🔄 Ldr	• È	<u>نا المجامعة المجامعة المحامة ا</u>
Mtga			
Bell Cica.hex			
File <u>n</u> ame:	".hex		<u>O</u> pen
Files of <u>t</u> ype:	hex files (.hex)	T	Cancel

Figure 2.10 Select Loader File Window

6. Select the proper HEX file (Gca.hex), then select **OK**.

The program will read the file and send the appropriate information to the accessory.

The Download Status area will reflect the status of the procedure. Then the message shown in the figure below will be displayed.

NOTE: You can pause and resume the operation at any time during the downloading procedure using the appropriate button. You can also end the procedure by closing the window.



Figure 2.11 Reset Accessory Message

7. After the software has been successfully loaded, press the Reset button on the side of the GCA top section, see Figure 2.9 for its location. This will retrieve the calibration information used for the GCA and reset the accessory. (You may need to turn the power to the GCA off, and then on again, if the Reset is not successful.)

The status message area on the **Main** window will display "Software Download Successful!"

8. Select the Exit button.

Installing the GCA

Changing the Settings

The Gas Cooling Accessory has been set up to run using the default settings used in the Accessory Loader program. Therefore, we recommend that you keep the settings as shown in the figure below.

To access the **Communications Settings** window, select the **Settings** button on the **Accessory Loader** window.

Communications Settings			
Port Number Data Bits COM1 Baud Rate 5 0 6 0 7 0 8 9600 9600 Parity None Mark 0 0dd Space Even	Handshaking DTR Control DTR Input CTS Output DSR Output DCD Output DSR Input RTS Input		
OK Cancel	Help		

Figure 2.12 Communications Settings Window

The table on the following page provides a brief explanation of the settings shown.

Table 2.2 Communications Setting Description Port The communications port Number used to connect an accessory to your controller. Select either COM 1 or COM 2 depending upon your system setup. Default setting = COM1 Data Bits Number of bits per character. Default setting = 8. Handshaking The required sequence of signals for communication between the GCA and the controller. $Default \ setting = DTR$ Control. Baud Rate The transmission rate for data signals. Default setting = 9600 Stop Bits The last element of a character that signals the end of the character. $Default \ setting = 1.$ (table continued)

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Settings

Installing the GCA

Table 2.2Accessory LoaderSettings (cont'd)

Setting	Description
Parity	A bit, transmitted before the stop bit, that is used to check for errors in communications. Default setting = None.

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Filling the GCA

Introduction

The GCA tank must be filled with liquid nitrogen before it can be used for cooling experiments with the DMA 2980. There are two methods you can use to fill the GCA, depending on your laboratory setup:

- If you have the available space and are able to keep a bulk storage reservoir near the instrument, you can use the *autofill* feature. This allows you to automatically refill the GCA with liquid nitrogen from your bulk storage reservoir, when the GCA is not actively cooling.
- If you must take the GCA to the bulk storage reservoir for refilling, you will need to use the *manual* method to fill the GCA tank with liquid nitrogen.

Refer to the appropriate section in this chapter for the method of filling desired.



Read the safety precautions for handling cryogenic materials (located in the prefix of this manual) before filling the GCA. Wear goggles or a face shield and gloves large enough to be removed easily whenever you handle liquid nitrogen.

This chapter also includes guidelines on when to use the GCA with your DMA instrument and basic operation of the GCA.

Autofilling the GCA

Autofilling refers to the automatic refilling of the GCA from the bulk storage tank, providing a readily available cooling source for experiments. This section tells you how to set up the GCA and the connected instrument to allow autofilling. See page 3-7 for information on manually filling the GCA.

The autofill feature ensures that a ready supply of liquid nitrogen is available for subambient experiments. It reduces the effort and time involved with refilling the GCA manually. Following is a list of the features associated with autofilling:

- It allows the bulk storage supply of liquid nitrogen to automatically refill the GCA on demand from the instrument.
- Typical fill times for automatic refilling will be approximately 20 minutes, if the tank is completely empty. The time for automatic refilling is much shorter (about 10 minutes), if there is any liquid nitrogen left in the tank.
- Any autofill cycle will automatically stop if a test is started on the instrument. Autofill can only occur when a method is <u>not</u> in progress.

For information on running subambient experiments refer to the appropriate chapter in the DMA 2980 operators manual.

Autofilling the GCA for the First Time

The GCA must be filled before cooling experiments can be performed on the DMA 2980. When you have completely installed the GCA as directed in Chapter 2, and are ready to use the autofill system, follow these steps: 1. Open the liquid feed valve on the low pressure [25 psi (175 kPa) maximum] bulk storage container. <u>Do not</u> close this valve again until the bulk storage container is empty, or wait until 15 minutes after the fill sequence has been completed. 2. Select Control/GCA fill on the Thermal Solutions DMA Instrument Control Main Menu. The GCA will be filled automatically. (See the Thermal Solutions User *Reference Guide* for further information.) The autofill will shut off when the dewar is full, the bulk storage tank is empty, or the GCA tank pressure is below one psi for more than one minute. NOTE: Cold gas will escape from the GCA vent valve and may escape from the relief valve under certain filling conditions. The fill process normally takes about 20 minutes. Frost will build up on the tubing and parts of the NOTE: GCA and storage tank while the liquid nitrogen is being transferred. The insulation on the bulk storage feed tube will become stiff and brittle during the autofill process. Allow the tube to return to room temperature before attempting to move or bend the tube.

Filling the GCA

Refilling the GCA After an Experiment

> To automatically refill the GCA with liquid nitrogen after an experiment is completed, you simply set the method-end conditions to select autofilling at the end of a method.

Manually Filling the GCA

NOTE:

The GCA is designed so that it can be filled *manually* as well as automatically. The automatic filling procedure has been discussed previously. The instructions found in this section explain the method used to manually fill the GCA. The manual fill mode should only be used when a bulk storage reservoir cannot be placed close to the GCA.

Follow the directions below to fill the GCA manually. Refer to Figures 3.1 and 3.2 for the location of the items called out in the procedure.

Adapters, which are not supplied, may be required.

- Make sure that the bulk storage source that will be used for filling the GCA is a low pressure [25 psi (175 kPa) maximum] container. Use a regulator if the pressure is greater than 25 psi (175 kPa).
- 2. Turn off the POWER switch on the GCA and disconnect the power cable (cable shown on Figure 3.1, page 3-8).
- 3. Disconnect the controller interface cable from the INSTRUMENT connection (connection shown on Figure 3.1, page 3-8).







4. Disconnect the coolant transfer tube from the GCA. This is the hose, shown in the figure below, which runs from the GCA to the instrument.



Figure 3.2 Manually Filling the GCA

	5.	Wheel the GCA to your bulk storage reservoir location.
	6.	Connect the bulk storage feed tube from the bulk storage reservoir to the N_2 Gas to Instrument/ LN ₂ Manual Fill fitting on the GCA (shown in Figure 3.2). Tighten all fittings.
	7.	Open the valve on the bulk storage reservoir and begin filling the GCA.
NOTE:	Col ma fillir abo	d gas will escape from the GCA vent valve and y escape from the relief valve under certain ng conditions. The fill process normally takes out 20 minutes.
NOTE:	Fro GC, beii sto dur ret mo	st will build up on the tubing and parts of the A and storage tank while the liquid nitrogen is ng transferred. The insulation on the bulk rage feed tube will become stiff and brittle ring the autofill process. Allow the tube to urn to room temperature before attempting to ve or bend the tube.
	8.	Fill the GCA with liquid nitrogen until it weighs 175 lbs. The tank will be about three-quarters full.
NOTE:	lf th will cor	ne GCA is overfilled, the excess liquid nitrogen automatically be boiled off when the GCA is nnected to the instrument.
	9.	Close the valve on the bulk storage reservoir. Allow time for the liquid within the transfer tube to evaporate (approximately 15 minutes).

	10. Disconnect the feed tube from the GCA.
♦ CAUTION:	Use care when wheeling the full GCA to another location. The agitation will cause increased venting of the liquid nitrogen, and it may tip over easily.
	11. Wheel the GCA back to the instrument and reconnect the coolant transfer tube from the instrument to the GCA.
	12. Reconnect the interface cable to the IN- STRUMENT connection and plug in the power cord. (See Figure 3.1 on page 3-8.)
	 Turn on the POWER switch. The GCA is ready to operate.

Operating Your GCA with the DMA

During experiments the DMA 2980 monitors the need for coolant and automatically communicates the need for the power to the GCA heaters. This enables the variation of the coolant flow, as needed, to obtain the set temperature required in your experiments. The operation of the DMA 2980 is controlled by both its keypad and the *Thermal Solutions* Instrument Control software.

The Gas Cooling Accessory is generally used with the TA Instruments DMA 2980 in the following situations:

- When a subambient heating segment is used that is slower than the intrinsic heating rate of the furnace (which is approximately 2-3°C/min.).
- When any cooling segment is used in a method.
- When an isothermal segment is used at a temperature below 100°C.

Even if you are not using the GCA for cooling, it will still be active so that the normal boil-off of the gas from the tank will purge the furnace and sample with dry nitrogen.

For more information on method segments, see the *Thermal Solutions User Reference Guide*. Filling the GCA

Basic Operation

Follow these basic steps to use the GCA with the DMA 2980.

- 1. Connect and fill the GCA as directed in this manual.
- 2. Mount the sample in the DMA clamp. See the *DMA 2980 Operator's Manual* for details.
- 3. Make sure the DMA power, DMA heater, and GCA power switches are switched on.
- 4. Use the *Thermal Solutions* software to set up and start your experiment. See the *Thermal Solutions User Reference Guide* for information.

Appendix A: Parts Lists

Service should only be performed by qualified service personnel. Please contact TA Instruments at one of the offices listed in Appendix B for service or replacement parts. To ensure that you receive the correct part for your unit, be sure to include the part number, description, instrument type, model number, and serial number.

Table A.1 GCA Parts List

Part No.	Description
264064.001	Gasket, Neoprene Tank, 3" Dia., GCA
991426.001	GCA Operator's Manual
991075.902	Tank, GCA Replacement
205225.037	Fuse
991441.001	Tube, Instrument Feed
991442.001	Tube, Tank Fill, GCA
270712.001	Cable, RS232
890035.901	Power Cord

Appendix A

Electrical Safety (repeated from Safety section)

	High voltages (120 Vac) are present in this instrument, only qualified service personnel should remove covers and make repairs.
! warning	The power at the instrument <i>must</i> be turned off, and the interface cable and power cord must be removed before any service or repair work is started.
	Hazardous voltage is present inside the GCA. Do not remove the clamp securing the cap to the dewar. There are no user-serviceable parts inside the GCA. Call TA Instruments for service.

Appendix B: Ordering Information

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

> HELPLINE—U.S.A. For assistance with thermal analysis applications, please call the Thermal Analysis Help Desk at 1-302-427-4070. SERVICE—U.S.A. For instrument service and repairs, please call 1-302-427-4050.

TA Instruments Ltd. Europe House, Bilton Centre Cleeve Road Leatherhead, Surrey KT22 7UQ England Telephone: 0-11-44-1372-360363 Fax: 0-11-44-1372-360135

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 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 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 Australia Unit 3 38-46 South Street Rydalmere NSW 2116 Autstralia 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

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