

GAS COOLING ACCESSORY for Discovery DMA 850 and DHR/AR Rheometer with EHP



Getting Started Guide

Revision P

Issued September 2018

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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
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Notes, Cautions, and Warnings

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

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

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

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-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2014/35/EU of THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 26 February 2014 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2004 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

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 2014/30/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 26 February 2014 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 1 - Basic immunity test requirements.

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 accessory 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 accessoire est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

There are several major areas of concern pertaining to personal safety when using the Gas Cooling Accessory. Please refer to the sections below.

Required Equipment

While operating this accessory, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

Accessory Symbols

The following label is displayed on the GCA for your protection:

Symbol	Explanation
<u>^</u>	This symbol indicates that you should read this Getting Started Guide in its entirety. This guide contains important warnings and cautions related to the installation, operation, and safety of the accessory. Ce symbole indique que vous devez lire entièrement ce guide de démarrage. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité de l'accessoire.

Please heed the warning labels and take the necessary precautions when dealing with these areas. The *Gas Cooling Accessory Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Handling Liquid Nitrogen

WARNING: 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.

AVERTISSEMENT: Manipulation sécurisée des matières cryogéniques

L'azote liquide est utilisé comme agent de refroidissement dans de nombreux essais d'analyse thermique. À cause de sa température extrêmement basse (-196°C), il peut brûler la peau. Vous devez être extrêmement prudent lorsque vous travaillez avec de l'azote liquide ou d'autres matières cryogéniques.

L'azote liquide peut:

- 1. provoquer des brûlures de la peau
- 2. remplacer l'air de la pièce dans laquelle vous vous trouvez
- 3. produire des pressions très élevées s'il est piégé dans les conduites ou les cuves.

The DMA 850 uses liquid nitrogen as a source of cold gas in the Gas Cooling Accessory (GCA). Because of its low temperature [-195°C (-319°F)], liquid nitrogen will burn the skin. When you work with liquid nitrogen, use the following precautions:

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

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

- 1 Wear goggles or a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.
- 2 Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.
- 3 The purity of liquid nitrogen decreases when exposed to air. If the liquid in a container has been open to the atmosphere for a prolonged period, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

WARNING: Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

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

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

AVERTISSEMENT: Asphyxiant potentiel

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

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

N'aérez pas le récipient accessoire de refroidissement de gaz (GCA) dans des espaces confinés.

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

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

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 above.

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.

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 Close the furnace.
- 2 Set temperature to -50°C.
- While the furnace is cooling, vapor should be coming from the DMA 850 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.
- Open the furnace a few minutes after it reaches -50°C. You should hear gas venting from the GCA. If you do not hear the gas venting, call TA Instruments for service.

WARNING: Always make sure the GCA system is installed correctly. Make sure the fill line from the bulk storage tank is hooked up properly.

AVERTISSEMENT: Assurez-vous toujours que le système GCA est correctement installé. Assurez-vous que la conduite de remplissage du réservoir de stockage en vrac est correctement connectée.

CAUTION: Never allow liquid nitrogen to be trapped in the fill line from the bulk storage tank to the GCA.

MISE EN GARDE: Ne laissez jamais l'azote liquide coincé dans la conduite de remplissage entre le réservoir de stockage en vrac et le GCA.

WARNING: Do not use high pressure bulk tanks [greater than 170 kPa gauge (25 psig)]. 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.

AVERTISSEMENT: N'utilisez pas de réservoir en vrac haute pression supérieur à 170 kPa (pression manométrique) (25 psig)]. Le GCA est conçu pour les réservoir en vrac basse pression L'utilisation de réservoirs en vrac haute pression entraîne le fonctionnement inapproprié du GCA et augmente le risque de blessures.

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.

MISE EN GARDE: Ne retirez jamais la conduite de remplissage automatique du GCA du réservoir de stockage en vrac sans fermer la vanne du réservoir d'abord.

When connecting and removing the GCA Autofill line remember to wear goggles and gloves.

WARNING: Do not use high pressure bulk tanks [greater than 170 kPa gauge (25 psig)]. 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.

AVERTISSEMENT: N'utilisez pas de réservoir en vrac haute pression supérieur à 170 kPa (pression manométrique) (25 psig)]. Le GCA est conçu pour les réservoir en vrac basse pression L'utilisation de réservoirs en vrac haute pression entraîne le fonctionnement inapproprié du GCA et augmente le risque de blessures.

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 (100–240 VAC) are present in this instrument, only qualified service personnel should remove covers and make repairs.

WARNING: The power at the instrument *must* be turned off, and the interface cable and power cord must be removed before any service or repair work is started.

AVERTISSEMENT: L'alimentation de l'instrument doit être coupée, le câble d'interface et le cordon d'alimentation doivent être retirés avant le démarrage des travaux de réparation ou d'entretien.

WARNING: 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.

AVERTISSEMENT: Présence d'une tension dangereuse dans le GCA. Ne retirez pas la bride de serrage qui maintient le bouchon au Dewar. Le GCA ne contient aucune pièce remplaçable par l'utilisateur. Appelez TA Instruments pour l'entretien.

Lifting the Instrument

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

WARNING: 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.

AVERTISSEMENT: Faites rouler le GCA sur ses roulettes pour le déplacer, si possible. Si vous devez le soulever, 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 GCA

Overview

The GCA (Gas Cooling Accessory) is a cooling accessory for use with the TA Instruments Discovery Series DMA 850 or with the DHR Series/AR-G2/AR 200ex/AR 1500ex Rheometer Electrically Heated Plates (EHP). The GCA has been designed for automatic refilling from a low pressure [170 kPa gauge (25 psig) maximum] bulk storage tank that can be located within 1.8 meters (6 feet) of the GCA.

The GCA is also capable of being filled manually or automatically when connected to a bulk storage source.



Figure 1 Gas Cooling Accessory.

NOTE: Before proceeding, be sure you understand and follow the safety precautions in the prefix of this manual.

Instrument Specifications

The specifications in the following tables apply to the Gas Cooling Accessory.

Table 1: GCA Technical Specifications

Item/Area	Specification
Liquid nitrogen capacity	50 L
Size	107 cm (42 in) high 46 cm (18 in) diameter of dewar 79 cm (31 in) diameter of feet
Weight	47 kg (104 lbs) empty 88 kg (194 lbs) full
Power requirements	100–240 VAC at 0.9 kVa, 47–63 Hz
Cooling capacity	-150°C (DMA 850), -70°C (EHP)
Pressure relief	90 kPa gauge (13 psig) relief valve on tank 345 kPa gauge (50 psig) on fill line
Liquid nitrogen tubes	Transfer: 1.8 meters (6 feet) insulated from GCA to instrument.
GCA fill modes	Automatic: Bulk storage within 1.8 meters (6 feet) of GCA. Manual: Remote filling at bulk storage location.
Bulk storage tank	Use low pressure supply tank only. Recommended filling pressure is 140 to 170 kPa gauge (20 to 25 psig)
Operating environment conditions	Temperature: 15–30 °C Conditions Relative Humidity: 5–80% (noncondensing) Installation Category II Pollution Degree 2 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 W heaters to vaporize the liquid nitrogen and obtain required pressures of up to 62 kPa gauge (9 psig). The pressurized gas is forced out of the tank and into the DMA 850 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 the figure below 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.

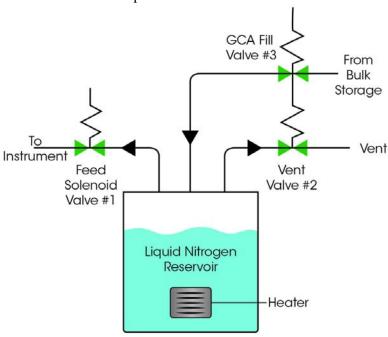


Figure 2 GCA operation illustration.

Description of Components

The following illustration shows the major parts of the TA Instruments GCA as it appears when connected to a DMA 850. The major parts of the GCA are the dewar, which holds the liquid nitrogen; the coolant transfer and feed hoses, which connect to the instrument and bulk liquid nitrogen source respectively; and the top section, which contains all of the ports and valves needed to make the connections.



Figure 3 Major GCA components.

Top Section of GCA

The top section of the GCA contains several items important to the operation of the accessory and the instrument. The figures below and on the next page illustrate the items found on sides of the GCA top section. (NOTE: The labeling of the GCA ports on the top section of your unit may vary slightly from the figure shown below.) Refer to the table on the next page for a description of the individual parts.



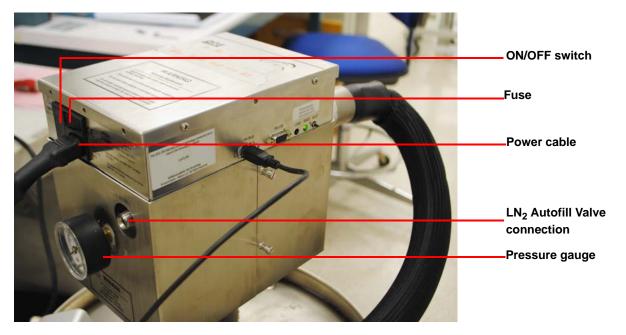


Figure 4 GCA top section.

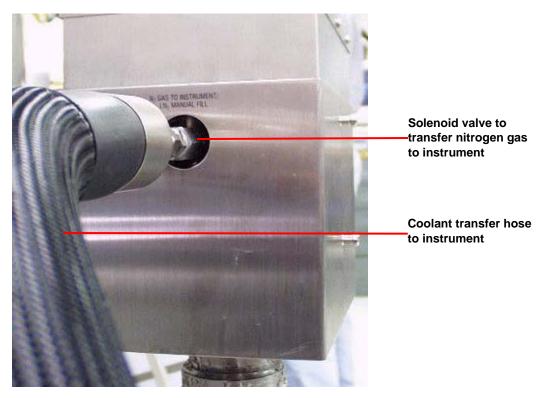


Figure 5 GCA top section.

Table 2: GCA Parts

Item/Area	Description
50 liter dewar	The thermally-insulated storage vessel for liquid nitrogen.
Coolant transfer hose	Supplies gaseous nitrogen from the GCA to the instrument.
Catch trough	Recessed area that allows condensation to collect on the top of the dewar. The resulting water can be released via a drain valve. See Chapter 2 for instruc- tions.
Interface cable	Provides the control signals from the instrument to the GCA.
Instrument/USB CAN BUS connection	For the DMA 850: Connects the GCA with the instrument to communicate information such as GCA tank pressure, heater regulation, etc. between the GCA and the instrument.
Accessory/RS-232 connection	For the DHR: Connects the GCA with the instrument to communicate information such as GCA tank pressure, heater regulation, etc. between the GCA and the instrument.

Item/Area	Description
Fill button	Used to automatically fill the GCA when it is disconnected from the instrument. See the DMA 850 online help 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.
ON/OFF switch	Turns the GCA power on. This switch must be on for the GCA to supply coolant to the instrument and to refill the GCA automatically from the bulk liquid nitrogen storage container.
Fuse	An 8A, 250V slow blow fuse is located in the top section of the GCA. If this fuse blows, you will get no response from the unit when you attempt to turn it on.
Power cable	Plugs into a source of electrical power to provide power for the operation of the GCA and its heaters.
LN ₂ Autofill Valve connection	Connect the GCA feed tube from this valve to supply liquid nitrogen from the bulk source to the GCA.
Pressure gauge	Provides a reading on the pressure contained within the dewar. This gauge should measure less than 90 kPa gauge (13 psig). The red line indicates the pressure at which the relief valve actuates.
Solenoid transfer valve	An automatic solenoid valve that opens to supply gaseous nitrogen to the instrument.
Bulk storage feed hose (not shown)	Allows the automatic and manual filling of the 50 L GCA dewar from a bulk storage source.

Chapter 2:

Installing the GCA

Unpacking and Inspecting

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:

WARNING: 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.

AVERTISSEMENT: Lisez les précautions de sécurité à prendre lors de la manipulation des matières cryogéniques (disponibles dans le préfixe du présent manuel avant d'installer le GCA. Portez des lunettes de protection ou un écran facial et des gants assez grands pour être retirés facilement chaque fois que vous manipulez de l'azote liquide.

• 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.



Figure 6 Clamp.

• Connecting the GCA to the DMA 850 requires a USB Type-B cable for connection, while the EHP employs an RS-232 cable. 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. The GCA should be:

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 (100–240 VAC, 50 or 60 Hz, 10 A). A step up transformer may be required to meet temperature specifications at line voltages lower than 120 VAC.
- your TA Instruments DMA 850.

Away from

- dusty environments.
- · 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.

Installing the GCA

The GCA can be connected to either a DMA850 or a DHR Rheometer with EHP system. Follow the instructions appropriate to your instrument.

Installing the GCA for use with a DMA 850

Installing the GCA primarily consists of connecting the interface cable and attaching the coolant transfer hose to the bulk liquid nitrogen source. Use the following basic steps to install the GCA.

- 1 Position the GCA within 1.8 meters (6 feet) of the instrument.
- 2 Attach the coolant transfer hose to the GCA at the fitting labeled N_2 Gas to Instrument/LN₂ Manual Fill.



Figure 7 Coolant transfer hose attached to the GCA.

Remove the air cool line (if present) from the DMA 850, and then attach the coolant transfer hose. Make sure the fittings are tight.

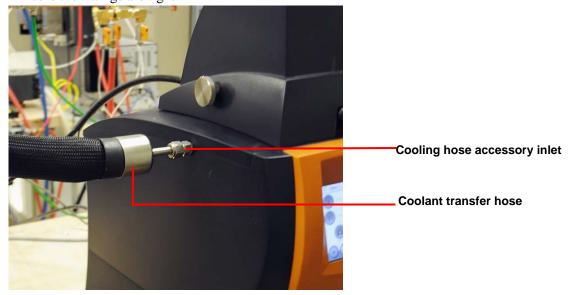


Figure 8 Connecting the coolant transfer tube to the DMA 850.

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

WARNING: The instrument power switch should be OFF before connections are made.

AVERTISSEMENT: Avant d'effectuer les raccordements, l'interrupteur d'alimentation de l'instrument doit être à l'arrêt.

- 5 Connect the USB CAN BUS cable from the DMA 850 to the GCA. See <u>Figure 4</u>.
- **6** Attach the power cable to the back of the GCA.

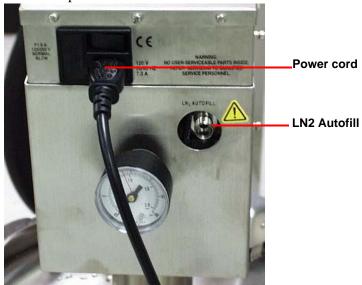


Figure 9 Power cord connected.

7 Turn the power switch to the ON position. When the ready light glows continuously, the accessory is

ready to be used.

The GCA is designed to be filled automatically from a *low pressure*, 140 to 170 kPa gauge (20 to 25 psig), bulk storage liquid nitrogen container.

If you will not be using the autofill feature, turn to <u>Chapter 3</u> for manual filling instructions.

To use the auto refill capability, follow the next several steps.

CAUTION: If your liquid nitrogen source has more than 170 kPa gauge (25 psig), then a pressure regulator must be added to insure no more than 170 kPa gauge (25 psig) is delivered to the GCA. Failure to limit the pressure may result in damage to the fill solenoid valve, cause excessive fill times, and cause the safety pressure relief valve to activate.

MISE EN GARDE: Si la source de votre azote liquide a une pression manométrique supérieure à 170 kPa (25 psig), alors vous devez ajouter un régulateur de pression pour vous assurer que la pression manométrique fournie au GCA ne dépasse pas 170 kPa (25 psig). Le non respect de cette limitation de pression peut endommager l'électrovanne de remplissage, prolonger à l'excès la durée de remplissage et provoquer l'activation de la soupape de détente de pression.

- 8 Arrange the low pressure bulk storage source physically close enough, within 1.8 meters (6 feet), to the GCA so that the autofill hose 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 1.8-meter (6-foot) coolant transfer hose.
- 9 Attach the bulk storage feed hose to the GCA at the connection labeled LN₂ Autofill as shown in <u>Figure</u> 9 above. Use a wrench to tighten the connector.

10 Attach the other end of the bulk storage feed hose assembly to the liquid feed connector on the bulk storage container.



Figure 10 GCA attached to DMA 850 (bulk source not shown).

Updating the GCA Software with the DMA 850

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 DMA 850 Online help topic "Updating Instrument Software Using TRIOS" to update the GCA program.

Turn to Chapter 3 for the instructions needed to fill the accessory with liquid nitrogen.

Installing the GCA for use with the DHR EHP System

The GCA can be used in conjunction with the Electrically Heated Plates (EHP) accessory on the DHR Series or AR-G2/2000ex/1500ex to provide cooling down to -70°C. Follow the instructions below to install the GCA.

- 1 Position the GCA within 1.8 meters (6 feet) of the instrument.
- 2 Attach the coolant transfer hose to the GCA at the fitting labeled N₂ Gas to Instrument/LN₂ Manual Fill.



Figure 11 Coolant hose attached to fitting.

Remove the crash cool air lines to the EHP (if present), and attach a short coolant transfer hose to the upper and lower coolant inlets as shown in the figure below. Make sure the fittings are tight.



Figure 12 Coolant transfer hose connected to upper and lower coolant inlets.

4 Connect the other ends of the short coolant transfer hoses and the main transfer hose from the GCA to the T-piece, as shown in the figure below. To avoid frosting during use, the T-piece can be insulated with pipe wrap. Connect the 8 mm FEP tubing to the upper EHP cooling exit pipe. This is required to match the back pressure of the upper cooling circuit to the lower, and also acts as an exhaust muffle.

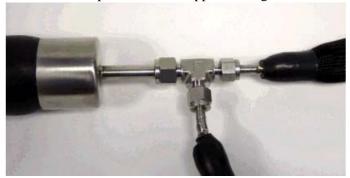


Figure 13 T-piece.

5 Make sure the power switch on the GCA is in the OFF position.

WARNING: The instrument power switch should be OFF before connections are made.

AVERTISSEMENT: Avant d'effectuer les raccordements, l'interrupteur d'alimentation de l'instrument doit être à l'arrêt.

6 Connect the interface cable between the RS-232 port on the GCA and RS232 AUX (for AR series) or RS232 (for DHR series) on the rheometer electronics box. See <u>Figure 14</u> and <u>Figure 15</u> below.

NOTE: Use the interface cable supplied with the GCA.

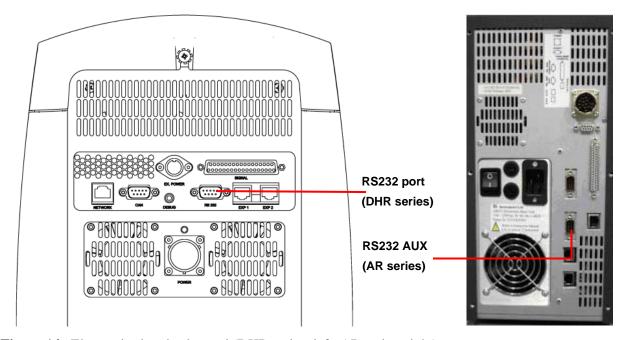


Figure 14 Electronics box back panel (DHR series, left; AR series, right).



Figure 15 GCA ports.

7 Attach the power cable to the back of the GCA.



Figure 16 Power cable attached.

- **8** Plug the power cable into a 100–240 VAC power source.
- 9 Turn the power switch to the ON position. When the ready light glows, the accessory is ready to be used.

NOTE: <u>Do not</u> plug the EHP in <u>before</u> powering on the GCA. Doing this will cause the GCA to be unrecognizable to the instrument and the GCA will not begin cooling. First power on the GCA and then plug in the EHP Smart SwapTM connector.

The GCA is designed to be filled automatically from a low pressure, 140 to 170 kPa gauge (20 to 25 psig), bulk storage liquid nitrogen container.

If you will not be using the autofill feature, turn to <u>Chapter 3</u> for manual filling instructions.

To use the auto refill capability, follow the next several steps.

For older GCA units that have two serial ports on the head, connect the interface cable supplied with the EHP cooling kit between COM A on the GCA and RS-232 AUS (for AR series) or RS-232 (for DHR series) on the rheometer electronics box.



Figure 17

CAUTION: If your liquid nitrogen source has more than 170 kPa gauge (25 psig), then a pressure regulator must be added to insure no more than 170 kPa gauge (25 psig) is delivered to the GCA. Failure to limit the pressure may result in damage to the fill solenoid valve, cause excessive fill times, and cause the safety pressure relief valve to activate.

MISE EN GARDE: Si la source de votre azote liquide a une pression manométrique supérieure à 170 kPa (25 psig), alors vous devez ajouter un régulateur de pression pour vous assurer que la pression manométrique fournie au GCA ne dépasse pas 170 kPa (25 psig). Le non respect de cette limitation de pression peut endommager l'électrovanne de remplissage, prolonger à l'excès la durée de remplissage et provoquer l'activation de la soupape de détente de pression.

- 10 Arrange the low pressure bulk storage source physically close enough, within 1.8 meters (6 feet), to the GCA so that the autofill hose 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 1.8-meter (6-foot) coolant transfer hose.
- 11 Attach the bulk storage feed hose to the GCA at the connection labeled LN₂ Autofill as shown in <u>Figure 9</u>. Use a wrench to tighten the connector.
- 12 Attach the other end of the bulk storage feed hose assembly to the liquid feed connector on the bulk storage container.

Refer to the next chapter for the instructions needed to fill the accessory with liquid nitrogen.

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 Swagelok® nut, screw the elbow into the fitting until it is hand tight with the valve pointing down.

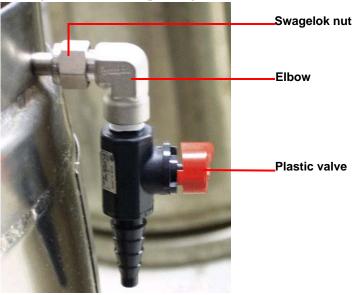


Figure 18 GCA condensate drain valve.

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.

MISE EN GARDE: Pendant les opérations de remplissage manuel, ne remplissez pas trop le réservoir GCA de manière à provoquer le déversement de l'azote liquide dans la cuvette de récupération.

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Chapter 3:

Operating and Maintaining the GCA

Overview

The GCA tank must be filled with liquid nitrogen before it can be used for cooling experiments. 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.

WARNING: 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.

AVERTISSEMENT: Lisez les précautions de sécurité à prendre lors de la manipulation des matières cryogéniques (disponibles dans le préfixe du présent manuel avant d'installer le GCA. Portez des lunettes de protection ou un écran facial et des gants assez grands pour être retirés facilement chaque fois que vous manipulez de l'azote liquide.

This chapter also includes guidelines on when to use the GCA with your DMA 850 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 32 for information on manually filling the GCA.

The autofill feature ensures that a ready supply of liquid nitrogen is available for subambient experiments as well as reducing the time and effort that is involved with manual filling of the GCA. The following is a list of 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 occurwhen a method is not in progress.

For information on running subambient experiments refer to the appropriate topic in Online help.

Autofilling the GCA for the First Time (DMA 850)

The GCA must be filled before cooling experiments can be performed on the DMA 850. 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 [170 kPa gauge (25 psig) maximum] bulk storage container. Do not close this valve again until the bulk storage container is empty, or wait until 15 minutes after the fill sequence has been completed.
- 2 Click the **GCA Fill** button in the TRIOS Control panel or press **GCA Autofill** on the DMA 850 user interface (located inside the Temperature screen) to start the automatic filling process.

The autofill will shut off when the dewar is full, the bulk storage tank is empty, or the GCA tank pressure is below one psig 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.

NOTE: Frost will build up on the tubing and parts of the 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.

Refilling the GCA After a DMA 850 Experiment

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

Autofilling the GCA for the First Time (DHR/AR)

The GCA must be filled before cooling experiments can be performed on the rheometer with the EHP installed. When you have completely installed the GCA, as directed in <u>Chapter 2</u>, and are ready to use the autofill system, follow these steps:

- 1 Open the liquid feed valve on the low pressure [170 kPa gauge (25 psig) maximum] bulk storage container. Do not 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 the **Set temperature system idle** icon.



Figure 19 Select Set temperature system idle.

NOTE: An autofill will also be initiated when the GCA is first powered on.

3 The autofill will shut off when the temperature system is activated by starting an experiment or setting a temperature on the status page, the dewar is full, the bulk storage tank is empty, or the GCA tank pressure is below one psig 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.

NOTE: Frost will build up on the tubing and parts of the 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.

Refilling the GCA after a Rheometer Experiment

To automatically refill the GCA with liquid nitrogen after an experiment is completed, select **Set temperature system idle** in the procedure post experiment step as shown in the figure below. If normal force control is not active in your experiment, it will necessary to manually activate the fill by selecting **Set temperature system idle** from the **Environmental** control panel.

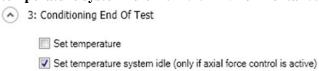


Figure 20 Set temperature system idle.

Autofilling the GCA Without the Instrument

The GCA can be autofilled while disconnected from the instrument.

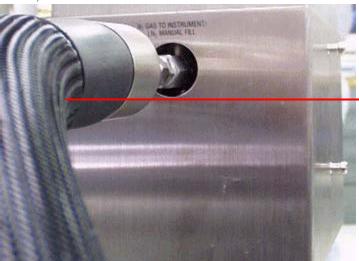
- 1 Attach the coolant transfer hose to the GCA at the fitting labeled N2 Gas to Instrument/LN2 Manual Fill.
- 2 Attach the power cable and make sure the power switch on the GCA is set to the **OFF** position.
- 3 Press the **Fill** button on the GCA. When filling stops automatically, within aproximately 20 minutes, the GCA is full.

Manually Filling the GCA

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 and no power source is available. Follow the directions below to fill the GCA manually.

NOTE: Adapters, which are not supplied, may be required.

- 1 Make sure that the bulk storage source that will be used for filling the GCA is a low pressure [170 kPa gauge (25 psig) maximum] container. Use a regulator if the pressure is greater than 170 kPa gauge (25 psig).
- 2 Turn off the POWER switch on the GCA and disconnect the power cable.
- 3 Disconnect the interface cable from the GCA. (See page 28 to determine the port used for your unit.)
- 4 Use a wrench to disconnect the coolant transfer hose from the GCA. This is the hose, shown in the figure below, which runs from the GCA to the instrument.



Remove this coolant transfer hose when manually filling the GCA. The bulk storage feed hose will be used here instead.

Figure 21 Disconnect the coolant transfer hose.

- 5 Wheel the GCA to your bulk storage reservoir location.
- 6 Connect the bulk storage feed hose from the bulk storage reservoir to the N₂ Gas to Instrument/LN₂ Manual Fill fitting on the GCA (shown in <u>Figure 21</u>). Tighten all fittings.
- 7 Open the valve on the bulk storage reservoir and begin filling the GCA.

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.

NOTE: Frost will build up on the tubing and parts of the 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.

8 Fill the GCA with liquid nitrogen until it weighs 175 lbs. The tank will be about three-quarters full.

NOTE: If the GCA is overfilled, the excess liquid nitrogen will automatically be boiled off when the GCA is connected to the instrument.

- 9 Close the valve on the bulk storage reservoir. Allow time for the liquid within the transfer hose to evaporate (approximately 15 minutes).
- 10 Disconnect the feed hose 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.

MISE EN GARDE: Soyez prudent lors du déplacement du GCA plein vers un autre emplacement. L'agitation fait augmenter l'évacuation de l'azote liquide et le GCA peut basculer facilement.

- 11 Wheel the GCA back to the instrument and reconnect the coolant transfer hose from the instrument to the GCA.
- 12 Reconnect the interface cable from the instrument to the GCA. (See page 28 to determine the port used for your unit.)
- 13 Turn on the POWER switch. The GCA is ready to operate.

Operating Your GCA with the DMA 850

During experiments the DMA 850 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 Gas Cooling Accessory is generally used with the TA Instruments DMA 850 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.

Basic Operation

Follow these basic steps to use the GCA with the DMA 850.

- 1 Connect and fill the GCA as directed in this manual.
- 2 Mount the sample in the DMA 850 clamp. See the appropriate documentation for instructions if needed.
- 3 Make sure the DMA 850 power, DMA 850 heater, and GCA power switches are switched on.
- 4 Use the instrument control software to set up and start your experiment.

NOTE: If you are starting your experiment at a subambient temperature, you may want to retighten the clamp at the lower temperature before beginning your run, depending on the clamp type. See the online help for details to perform this step.

Maintaining the GCA

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 instrument, untrained personnel must not attempt to test or repair any electrical circuits.

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

The Gas Cooling Accessory actually requires very little maintenance. The following items may need attention and are covered in this section:

- Cleaning
- Fuse replacement

Cleaning the GCA

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

WARNING: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the unit.

AVERTISSEMENT: N'utilisez pas de produits chimiques agressifs, de nettoyants abrasifs, de la laine d'acier ou tout autre matériau rugueux pour nettoyer l'appareil.

Replacing the Fuses

You can replace the fuses found in the power entry module located on the rear of the electronics control box. To check or change these fuses follow the instructions below and refer to the figures as needed:

- 1 Turn the cooling accessory off and remove the power cord.
- 2 Insert a small screwdriver on the edge of the fuse drawer to pull it out of the instrument.

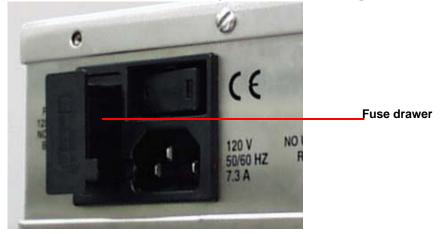


Figure 22 Fuse drawer.

3 Slide the fuse holder out of the fuse drawer.

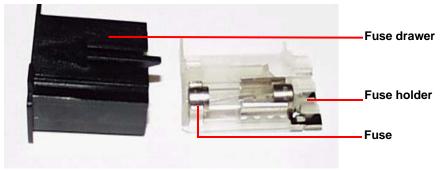


Figure 23 Fuse drawer with fuse and fuse holder.

- 4 Remove the old fuse and replace the fuse only with the type and rating indicated on the instrument's rear panel.
- 5 Place fuse holder back into the fuse drawer and slide the drawer into the opening.
- 6 Replace the power cord and turn the unit back on.

Replacement Parts

Service should only be performed by qualified service personnel. Please contact TA Instruments at one of the offices listed in the TA Manual Supplement. See the link on page 3 for information. 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 3: GCA Parts List

Part No.	Description
264064.001	Gasket, Neoprene Tank, 8 cm (3 inch) diameter, GCA
991075.902	Tank, GCA Replacement
201969.002	Fuse
991441.001	GCA/Bulk Supply LN ₂ Transfer Line
991442.001	DMA/GCA LN ₂ Transfer Line
265270.001	USB Cable
270712.002	RS-232 Cable, 10' long
253827.000	DMA Power Cord, NEMA 5–15