

# NITROGEN PURGE COOLER ACCESSORY for the Discovery DMA 850



### **Getting Started Guide**

#### **Notice**

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### Introduction

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

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

- 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.

#### **Safety**

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

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

#### **Instrument Symbols**

The following labels are displayed on the Nitrogen Purge Cooler for your protection:

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the NPC.  Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obtenir d'importantes informations relatives à sécurité. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité du système NPC.

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

#### **Chemical Safety**

WARNING: Do not use hydrogen or any other explosive gas in the NPC.

AVERTISSEMENT: N'utilisez pas d'hydrogène ou tout autre gaz explosif dans le NPC.

WARNING: If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

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

WARNING: Air is not to be used as the purge gas with this cooler. Oxygen will be condensed into liquid and pushed into the DMA furnace.

AVERTISSEMENT: l' Air ne doit pas être utilisé les gaz de drainage comme avec ce refroidisseur. L'Oxygène se condensera en liquide et sera poussé dans le four DMA

WARNING: MAY CAUSE SEVERE FROSTBITE. Can cause severe frostbite to the eyes and skin. Do not touch frosted pipes or fittings.

AVERTISSEMENT: Peut causer de graves gelures. Peut causer des gelures graves aux yeux et à la peau. Ne pas toucher les tuyaux ou les raccords givrés.

WARNING: Do not overfill the Dewar. Leave an inch of space from the top of the Dewar when filling. Liquid nitrogen may boil over the sides when the Cooling Gas is turned on.

AVERTISSEMENT: Ne surchargez pas le Dewar. Laisser un espace d'un pouce à partir du haut du Dewar lors du remplissage.

#### **Handling Liquid Nitrogen**

This cooling accessory uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature (-196°C [-321°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 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.

**NOTE**: Using the NPC poses all of the same risks as with any open Dewar of cryogenic liquid. Safe practices concerning the filling and transporting of small Dewars and use of Personal Protective Equipment is necessary.

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

### WARNING: Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent the Nitrogen Purge Cooler (NPC) 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 NPC 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.

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## Chapter 1:

### Introducing the Nitrogen Purge Cooler

### Overview

The Nitrogen Purge Cooler (NPC), is an optional accessory for extending the temperature range of the Discovery DMA 850 standard furnace to -160°C. The NPC consists of a 2.5 L Dewar flask that contains a copper coil tube. The furnace is cooled by purging nitrogen gas through the copper coil immersed in the liquid nitrogen-filled Dewar flask. The NPC requires a nitrogen gas supply (25 to 120 psi) at a flow rate of 30 LPM, access to a supply of liquid nitrogen, and equipment necessary for safe handling, transportation, and pouring of the liquid.



**Figure 1** NPC with DMA 850 instrument.

**NOTE**: The NPC is for crash cooling and controlled heating only. Controlled cooling rates are possible using the Gas Cooling Accessory (GCA), or Air Chiller Systems (ACS-3 and ACS-2).

### System Components

The NPC Accessory has the following major hardware components:

- A Dewar flask with insulated lid, capable of holding approximately 2.5 L of liquid nitrogen, and with a
  precise length of copper tubing to adequately chill the nitrogen purge gas.
- A length of 1/8" OD tubing that connects the NPC to the Cooling Gas Outlet fitting on the rear of the DMA.

### Accessory Specifications

The tables below detail the NPC Accessory technical specifications.

**Table 1: NPC Accessory Characteristics** 

Dimensions of Dewar flask	Height: 15 in. Width: 12 in. Depth: 7 in. Volume: 2.5 L
Weight of Dewar flask	6 lbs (without liquid nitrogen)

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

Gas	Nitrogen ONLY
Pressure	25–120 psi
Flow Rate	30 LPM

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

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

#### **Specifications**

Lowest temperature	-160°C
Time to reach low temperature	40 min
Time to reach -150°C	35 min
Heating rate: Minimum <sup>a</sup>	3°C/min

a. With NPC off and dependent on ambient temperature.

## Chapter 2:

### Installing the Nitrogen Purge Cooler

### Unpacking/Repacking the NPC

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

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

### Preparing the Accessory

Before shipment, the NPC is inspected so that it is ready for operation upon proper installation. Installation involves the following procedures:

- Inspecting the accessory for shipping damage and missing parts
- Connecting the NPC tubes and fittings
- Filling the Dewar

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

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

#### **Inspecting the System**

When you receive the NPC Accessory, look over the accessory and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

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

#### **Choosing a Location**

Choose a location for the accessory using the following guidelines. The NPC Accessory should be:

#### In

- A temperature-controlled area. Temperatures should be in the range of 20–30°C.
- A clean environment, preferably on the ground floor in the building.
- An area with ample working and ventilation space.

#### On

A stable work surface.

#### Near

Your DMA.

#### Away from

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

### Connecting the NPC to Your Discovery DMA

To connect the NPC, access the rear panel of the DMA and follow the instructions below:

**NOTE**: Remove the foam insulation from the Dewar before making connections and filling the Dewar with nitrogen.

- 1 Place the Dewar next to the DMA.
- 2 Disconnect the air cool line on the DMA.



Figure 1 Disconnect the air cool line.

3 Remove the cooling gas tubing from the COOLING GAS OUTLET port on the back of the DMA.



Figure 2 Disconnect cooling gas tubing.

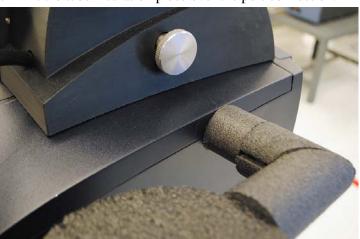
4 Connect the copper tubing from the NPC to the cooling gas port on the DMA. Take care to position the

NPC to minimize the strain at the connection between the NPC and the cooling gas port.



Figure 3 Connect copper tubing to the DMA.

5 Fit the tube insulation piece over the port connection.



**Figure 4** Tube insulation piece covering the port connection.

6 Connect the nitrogen purge gas tubing to the COOLING GAS OUTLET port on the back of the DMA. Outlet pressure is internally regulated to 20 psig; no adjustment is necessary. Use nitrogen gas ONLY.

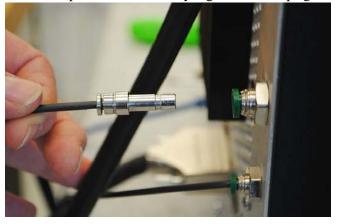


Figure 5 Nitrogen purge gas tubing and COOLING GAS OUTLET.

7 Connect the nitrogen gas source to the COOLING GAS INLET port on the back of the DMA 850. The inlet gas pressure range is 25–120 psig at a flow rate of 30 L/min.

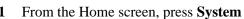
**NOTE**: The 20" length of 1/8" tubing is critical. Do not shorten this tube. Shortening this tube can cause an excessive amount of nitrogen to be condensed into liquid and pushed into the furnace.

8 Proceed to the <u>next section</u> to set up the accessory in TRIOS. Do not <u>fill the Dewar</u> until the next section has been completed.

### Setting up the NPC Accessory using TRIOS

CAUTION: Operation of this accessory is intended for the DMA 850 with instrument software version 4.3 or higher only.

After successfully installing the NPC, set up the accessory from the DMA 850 user interface.

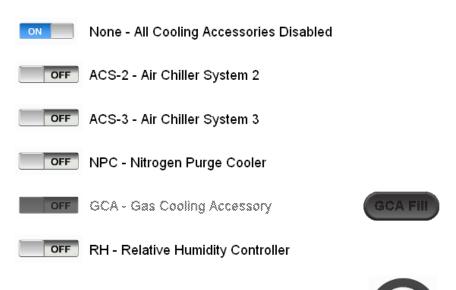




The System screen displays.

- 2 From the System screen, press **Cooling Accessories** . The Environmental Accessory selection screen displays.
- 3 Press the **OFF** button next to **NPC Nitrogen Purge Cooler** to turn the accessory on.

Select Environmental Accessory:



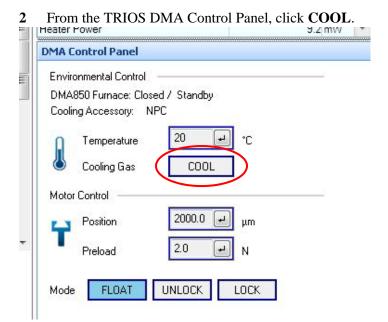


4 Proceed to Filling the Dewar.

### Filling the Dewar

Before the Dewar can be filled with liquid nitrogen, the copper tube must be purged with dry gas. This is necessary to prevent moisture from freezing in the copper tube when liquid nitrogen is added to the Dewar; the frozen moisture blocks the flow of the cooling gas.

1 Make sure the furnace is closed.



**Figure 6** Turn on the Cooling Gas in the software.

- 3 Allow the dry gas to purge through the copper tube for at least 20 minutes, or until the copper tube is dry.
- 4 Turn the Cooling Gas off (the same way it was turned on in the software).
- 5 Fill the Dewar with liquid nitrogen.

WARNING: Do not overfill the Dewar. Liquid nitrogen may boil over the sides when the Cooling Gas is turned on.

AVERTISSEMENT: Ne pas trop remplir le Dewar. L'azote liquide peut avoir une ébullition sur les côtés lorsque le gaz de refroidissement est activé.

## Chapter 3:

### Use and Maintenance

### Using the NPC Accessory

All of your NPC Accessory experiments will follow the same general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. See the instrument control online help for instructions on performing these actions.

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

Please note the following when performing an experiment:

- One full Dewar (approximately 2.5 L of liquid nitrogen) will typically be consumed for each experiment.
- The nitrogen gas for the NPC is supplied by a connection to the Cooling Gas Inlet port on the back of the DMA 850. If the source of the gas is a high pressure, compressed nitrogen supply at approximately 2000 psig, seven complete experiments as shown above can be performed.

#### **Before You Begin**

Before using the NPC Accessory, ensure that the is installed properly. Also make sure you have:

- Connected the NPC
- Powered on the DMA
- Filled the Dewar as necessary
- Specified the NPC in the instrument control software
- Become familiar with controller operations

It is a good idea to purge the copper coil with dry nitrogen just prior to filling the Dewar with liquid nitrogen. Select **Control** > **Cooling Gas** > **On** to purge the coil with dry nitrogen. After a few minutes, select **Control** > **Cooling Gas** > **Off** to turn the purge gas off again.

### Maintaining the Accessory

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel.

#### **Cleaning the Accessory**

To clean the NPC Accessory, wipe down the exterior of the Dewar with a damp, soft cloth.

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

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

### Replacement Parts

**Table 4: Replacement Parts for the NPC Accessory** 

Part Number	Description
986316.901	Insulation Replacement Kit NPC