# LN2P

# Liquid Nitrogen Pump System



**Getting Started Guide** 



#### **Notice**

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

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## Introduction

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

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

- TA Instruments Trademarks
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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.

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**

#### For Canada

CAN/CSA-22.2 No. 61010.1-04 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-22.2 No.61010.2.010-04 Particular requirements for laboratory equipment for the heating of materials.

#### For European Economic Area

EN61010-1: 2010 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part I: General requirements.

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

#### 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

EN61326-1: 2006 Electrical equipment for measurement, control, and laboratory use - EMC requirements - Part 1: General requirements, Table 1 - Basic immunity test requirements, Emission requirements for Group 1, Class A equipment.

#### 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**

### **Instrument Symbols**

The following label is displayed on the LN2P for your protection:

Symbol	Explanation	
<u> </u>	This symbol on the LN2P 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 LN2P.	
	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 la LN2P.	
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface.	
<u> </u>	Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.	

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

WARNING: Due to the size and weight of the cooling accessory, the LN2P should always be lifted by two people and only when empty to prevent injury.

AVERTISSEMENT: En raison de la taille et du poids de l'accessoire de refroidissement, le LN2P doit toujours être soulevé par deux personnes et à vide uniquement pour éviter des blessures.

WARNING: The cooling head assembly contains coated Fiberfrax material. Excessive handling of this material could cause Fiberfrax particles to be emitted into the air. See the MSDS sheet for safety measures to be observed when Fiberfrax is used.

AVERTISSEMENT: L'ensemble de la tête de refroidissement contient un revêtement en Fiberfrax. La manipulation excessive de ce revêtement pourrait entraîner l'émission de particules de Fiberfrax dans l'air. Voir la fiche technique santé-sécurité pour les mesures de sécurité à observer en cas d'utilisation du Fiberfrax. WARNING: The LN2P is only to be used with the supplied power supply, P/N 202084.001.

AVERTISSEMENT: Le LN2P doit être utilisé uniquement avec l'alimentation électrique fournie, N° de pièce 202084.001.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

### Handling Liquid Nitrogen

The LN2P uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. 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.

- 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.
- 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.
- 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.

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 Liquid Nitrogen Pump System (LN2P) 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 système de pompage de l'azote liquide (LN2P) 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.

## Thermal Safety

The cell surfaces can be hot enough to burn the skin during a sample run. If you are conducting a subambient test on the Discovery DSC, cold could also cause injury. After running any type of experiment, you must allow the Discovery DSC cell to return to room temperature before you touch the inner cell surfaces.

WARNING: Some surfaces of the LN2P and Discovery DSC system may get extremely cold when using the LN2P for cooling experiments. This presents a danger to exposed skin coming in contact with and adhering to the cold surfaces. To prevent moisture buildup in the system, we recommend that you do not remove the DSC lid when the instrument is at subambient temperatures. However, if you do remove the lid or handle any cold surfaces, use forceps or gloves to prevent injury.

AVERTISSEMENT: Certaines surfaces du LN2P et du système Discovery DSC peuvent devenir extrêmement froides lors de l'utilisation du LN2P pour des expériences de refroidissement. Cela représente un danger pour les peaux exposées qui entrent en contact avec les surfaces froides et y adhèrent. Pour éviter l'accumulation de la moisissure dans le système, nous recommandons de ne pas retirer les couvercles du DSC lorsque l'instrument est à basse température. Cependant, si vous retirez le couvercle ou manipulez des surfaces froides, utilisez des pinces ou des gants pour éviter des blessures.

WARNING: When performing the remote fill procedure described on page 33, always ensure that the manual cap fittings are securely in place on the supply and return lines BEFORE filling the LN2P. If the supply and return lines are not capped, they will discharge liquid nitrogen during remote filling.

AVERTISSEMENT: Lorsque vous effectuez la procédure de remplissage à distance décrite en page 33, assurez-vous toujours que les raccords manuels de capuchon sont solidement installés sur les conduites d'alimentation et de retour AVANT de remplir le LN2P. Si les conduites d'alimentation et de retour ne sont pas fermées, elles vont libérer de l'azote liquide pendant le remplissage à distance.

#### Water Condensation

WARNING: Some of the DSC and LN2P surfaces get cold during use of the LN2P. The cold surfaces can cause condensation and, in some cases, frost can build up. This condensation may drip to the floor. Make provisions to ensure the floor dry stays dry. A slipping hazard may result if the condensation is not cleaned up.

AVERTISSEMENT: Certaines surfaces du DCS et du LN2P deviennent froides pendant l'utilisation du LN2P. Certaines surfaces froides peuvent provoquer la condensation et dans certains cas, le givre peut s'accumuler. Cette condensation peut s'écouler et toucher le sol. Prenez des dispositions pour vous assurer que le sol reste sec. Si la condensation n'est pas nettoyée, il peut en résulter un risque de dérapage.

### Temperature Range

CAUTION: Do not exceed 100°C with the LN2P cooling head installed and the LN2P not being enabled. Serious damage to the cooling head could occur.

MISE EN GARDE: Ne dépassez pas 100°C lorsque la tête de refroidissement du LN2P est installée et le LN2P n'est pas activé. Cela pourrait provoquer de graves dégâts à la tête de refroidissement.

CAUTION: We recommend that you do not use the LN2P when running isothermal experiments above 400°C. The life of the DSC cell heating element can be shortened if the LN2P is used at high temperatures for extended periods.

MISE EN GARDE: Nous recommandons de ne pas utiliser le LN2P lorsque vous effectuez des expériences isothermes supérieures à 400°C. La durée de vie de l'élément chauffant de la cellule DSC peut être réduite si le LN2P est utilisé à des températures élevées sur de longues périodes.

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# Chapter 1:

## Introducing the LN2P

## Overview

The LN2P (Liquid Nitrogen Pump) is a cooling accessory for use with the TA Instruments Discovery DSC<sup>TM</sup>. The LN2P allows continuous temperature control within the range of -180 to  $550^{\circ}$ C. The LN2P delivers (via a pump) liquid nitrogen to the heat exchanger, which in turn cools the cell.

The LN2P can be controlled from the **General** panel of TRIOS software or the Common Cabinet User Interface.



Figure 1 LN2P with Discovery DSC.

## Components

The LN2P is made up of a 50-liter Dewar, the motor housing, and a cooling head that is connected to the Dewar by a 1.8-meter (6-foot) long supply/return hose. Components are shown in the figure below.

There are three plumbing fittings that you can access for normal operation:

- The first two fittings are for connection of the cooling head and feed/exhaust line. They are located within the motor housing. One fitting (1/4-inch tube) is for the liquid nitrogen supply to the heat exchanger (in the cooling head), and the other fitting (3/8-inch tube) is for the return liquid from the heat exchanger. For instructions on attaching these lines, see Chapter 2.
- There is an LN2 fill valve on the rear of the motor housing for connection to a bulk LN2 source. This fitting is for filling purposes and is controlled by a solenoid valve. This fitting is used for both autofilling and remote filling. For instructions on the use of this port, see <a href="#">Chapter 3</a>.

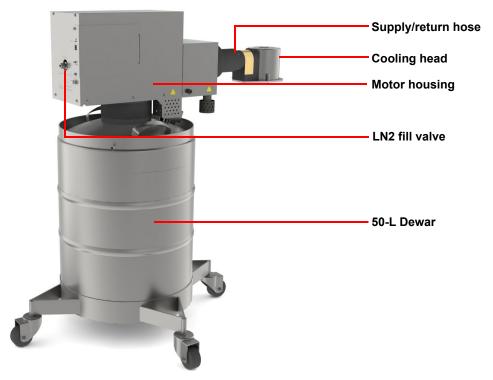


Figure 2 LN2P components.

## Instrument Specifications

The table found below contains the technical specifications for the LN2P.

**Table 1: LN2P Technical Specifications** 

Item/Area	Specification	
Instrument compatibility	Discovery DSC	
Size Height Width Depth Weight	107 cm (42 in) 86 cm (34 in) 86 cm (34 in) 50 kg (110 lbs) empty	
Power requirements for power adapter and pump inlet	92.5 kg (204 lbs) full  100–240 VAC / 2A, 50/60Hz (Pump inlet 24VDC, 2.5A)	
Controlled temperature range	−180 to 550°C	
Pressure relief	90 kPa gauge (13 psig) for Dewar 345 kPa gauge (50 psig) for fill line	
Pressure regulator	14 kPa gauge (2 psig) for pressure regulation	
Liquid nitrogen feed hose	1.8 m (6 ft) insulated from LN2P to heat exchanger	
Liquid nitrogen fill hose	1.8 m (6 ft) insulated from LN2P to bulk storage. Supplied with union and adapter for bulk storage connection. Do not use other lines to fill the LN2P, as lower flow resistance will increase Dewar pressure.	
Bulk storage tank	Use low pressure bulk supply tank only. Recommended source pressure is 140 to 170 kPa gauge (20 to 25 psig)	
Operating environment conditions	Temperature: 15–35°C 5% to 80% RH from 15°C to 31°C, decreasing to 66% RH at 35°C (non-condensing) Installation Category II Pollution Degree 2 Maximum Altitude: 2000 m (6560 ft)	

CAUTION: We recommend that you do not use the LN2P when running isothermal experiments above 400°C. The life of the DSC cell heating element can be shortened if the LN2P is used at high temperatures for extended periods.

MISE EN GARDE: Nous recommandons de ne pas utiliser le LN2P lorsque vous effectuez des expériences isothermes supérieures à 400°C. La durée de vie de l'élément chauffant de la cellule DSC peut être réduite si le LN2P est utilisé à des températures élevées sur de longues périodes.

# Chapter 2:

## Installing the LN2P

## Unpacking and Inspecting the System

Inspect the contents of the LN2P shipping box. You should retain the shipping container and packing materials at least until the unit has been successfully installed and verified to be functioning correctly, and you may wish to retain them in case you want to repack and ship your LN2P.

If the LN2P 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 accessory until an authorized representative of TA Instruments has repaired it.

Contact your TA Instruments representative if parts are missing.

## Before Installing the LN2P

Installation of the LN2P is generally the same for all types of DSC instruments.

WARNING: Read the safety precautions for handling cryogenic materials (located in the safety section of this manual) before filling the LN2P. Whenever you handle liquid nitrogen, wear goggles or a face shield and gloves large enough to be removed easily.

AVERTISSEMENT: Lisez les précautions de sécurité à prendre lors de la manipulation des matières cryogéniques (disponibles dans le section sécurité du présent manuel) avant de remplir le LNCS. 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.

WARNING: Unplug the power cord before beginning any service or repair work.

AVERTISSEMENT: Débranchez le cordon d'alimentation avant de commencer des travaux d'entretien ou de réparation.

WARNING: The LN2P is only to be used with the supplied power supply, P/N 202084.001.

AVERTISSEMENT: Le LN2P doit être utilisé uniquement avec l'alimentation électrique fournie, N° de pièce 202084.001.

## **Choosing a Location**

Because of the sensitivity of experiments using the LN2P, it is important to choose a location using the following guidelines. Refer to the *Discovery DSC Getting Started Guide* for more detailed information. Your LN2P should be:

#### In

- A temperature-controlled area.
- A clean environment.
- An area with ample working and ventilation space. Refer to the technical specifications in Chapter 1 for the accessory's dimensions.

#### Near

- A power outlet (100–240 Vac, 50 or 60 Hz).
- Your TA Instruments thermal analysis controller computer and Discovery DSC.

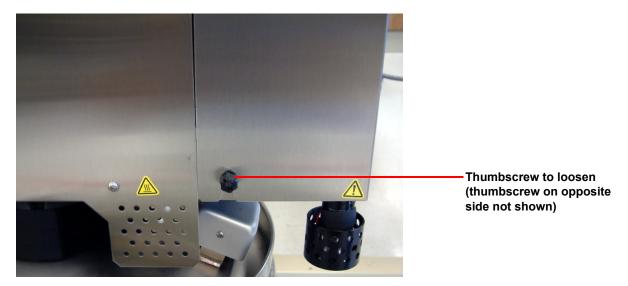
## Away from

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.

## **Installing the Transfer Line**

Before the LN2P can be installed on the Discovery DSC, the transfer line must first be attached to the LN2P. Follow the instructions below to install the transfer line.

1 Loosen the two thumb screws on either side of the front of the motor housing. Lift the cover up and away from the LN2P to remove it.



**Figure 3** Thumbscrew on motor housing.

2 Remove the Velcro strap from around the insulation block.

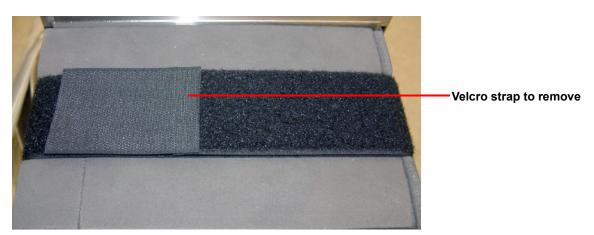


Figure 4 Velcro on insulation block.

3 The insulation block has an opening on one side, and a hinge on the other. Carefully open the insulation block to expose the two fittings that will be attached to the feed hose, and remove the insulation block from the LN2P.

4 Using 9/16-inch and 11/16-inch wrenches, remove the manual fill cap fittings from the liquid delivery lines and allow them to hang from their tethers.

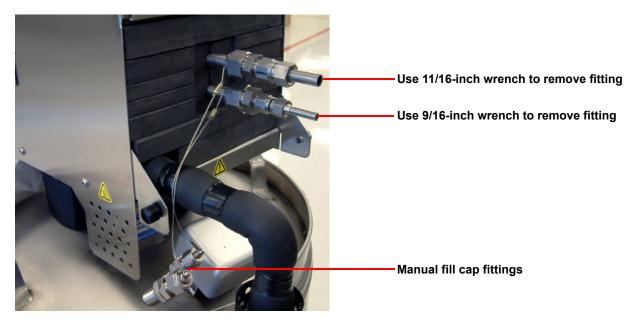


Figure 5 Manual fill cap fittings removed from liquid delivery lines.

5 Obtain the 1.8-m (6-foot) long feed line with its attached cooling head. At the opposite end from the cooling head are two fittings that need to be connected to the LN2P tank. The figure here identifies the two fittings that will be attached.



**Figure 6** Two fittings to connect to LN2P tank.

- **6** Connect the transfer line to the LN2P:
  - **a** Using a 9/16-inch wrench, attach the smaller liquid supply line to the smaller fitting.
  - **b** Using an 11/16-inch wrench, attach the larger return line to the remaining fitting as shown in the figure below.

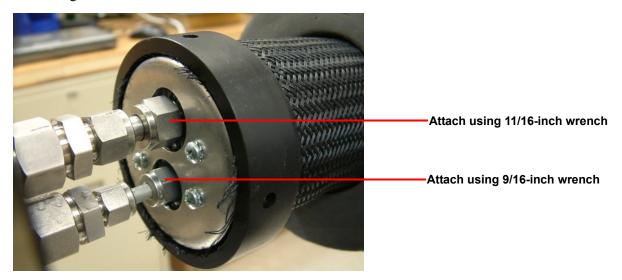


Figure 7 Transfer line connected to LN2P fittings.

- 7 Carefully replace the insulation block in the reverse method that was used to remove it.
- 8 Place the manual fill cap fittings in the insulation block.



Figure 8 Manual fill cap fittings in insulation block.

9 Replace the front of the motor housing, making sure its top lip slides into the receiver groove on the motor housing sheet metal. Tighten the thumb screws on either side finger tight.

## **Installing the LN2 Pump to the Tank**

Ice and frost are created during normal use of the LN2P. To prevent system failure, ensure inside of the tank, pump, and level sensor are clean with no moisture prior to attaching the pump to the tank.

When attaching the pump to the tank, align supply and return lines centered to the vacuum seal cap before installation of the clamp.

CAUTION: Incorrect alignment of the pump on the tank may cause damage to the vacuum seal cap. To avoid risk of freezing the vacuum seal cap, Figure 9 shows the orientation of pump to tank.

MISE EN GARDE: Un mauvais alignement de la pompe sur le réservoir peut endommager le bouchon d'étanchéité sous vide. Pour éviter le risque de gel du capuchon d'étanchéité à vide, la figure 9 montre l'orientation de la pompe vers le réservoir.

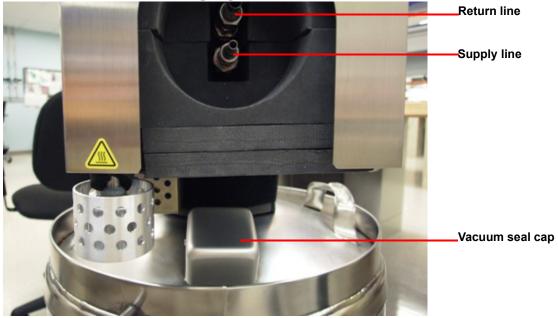


Figure 9 Pump and tank orientation.

## Installing the LN2P on the Discovery DSC

Installation of the LN2P onto the Discovery DSC involves:

- 1 Removing the Discovery DSC covers
- 2 Mounting the cooling head
- 3 Connecting the heater cable
- 4 Replacing the covers
- 5 Connecting the cables and lines

This section provides the instructions for installing the LN2P on the Discovery DSC.

## **Mounting the Cooling Head**

The LN2P unit connects to the DSC cell via a cooling head. This cooling head is made up of an internal heat exchanger, anti-condensate heaters, and various other components. The following steps describe the procedure for making the connection between the DSC and the LN2P unit.

- 1 Ensure the cell is at a temperature where contact with the cell will not cause a burn.
- 2 Press the Lid button on the Discovery DSC keypad to close the AutoLid, as shown in <u>Figure 10</u>.
- 3 Remove the three top-cover screws circled in the figure below.



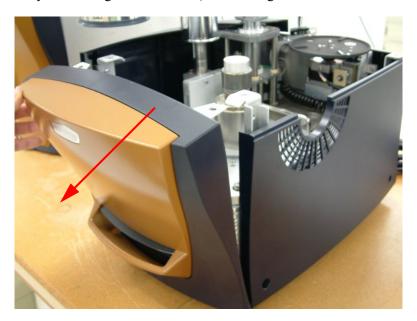
Figure 10 Discovery DSC top cover.

- 4 Press the Lid button on the DSC keypad to open the AutoLid on your instrument.
- 5 Remove the Autosampler tray and Autosampler sample waste bin from the top cover. Refer to Figure 10 for locations.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

- 6 Gently raise and rotate the Autosampler arm to the left (when facing the instrument) so that Autosampler cover and arm do not impede removal of the top cover.
- 7 Carefully lift up and pull the top cover away from the Discovery DSC (to the right when facing the instrument).
- 8 Once the top cover is removed, remove the front cover by pulling it away from the instrument (towards you if facing the instrument). See the figure below.



**Figure 11** Removing the front cover.

9 Verify that the DSC cell surface (indicated in the figure below) on the top of the silver block is not damaged or compromised. If any irregularities such as dents, buildup, contaminants, or oxidation are observed in the silver, contact your local TA Instruments Service Representative for details on re-dressing the cell.



Figure 12 Surface of silver block.

- 10 Verify that the DSC cell cooling flange is not damaged or compromised. Also inspect the graphite gasket that is installed on the mating surface of the copper heat exchanger in the LN2P cooling head. If any irregularities such as dents, buildup, contaminants, or oxidation are observed, contact your local TA Instruments Service Representative for details on re-dressing the flange.
- 11 Position the LN2P in close proximity and to the right of the DSC.

CAUTION: Take extreme care not to damage the silver block shown in <u>Figure 12</u>. The silver surfaces on the cell are soft and, if damaged, must be replaced by a TA Instruments Service Representative.

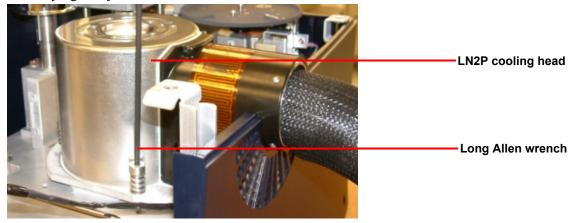
MISE EN GARDE: Soyez extrêmement prudent pour ne pas endommager le bloc en argent illustré sur la Figure 11. Les surfaces en argent de la cellule sont douces; si elles sont endommagées, faites les remplacer un représentant du service d'entretien de TA Instruments.

12 Align the pin on the cell base (shown in the figure below) with the corresponding slot in the LN2P cooling head and carefully lower the cooling head over the cell. Be particularly careful to avoid bumping the top surface of the cell with the cooling head, as any damage to the cell surface could adversely affect subsequent performance.



Figure 13 Exposed DSC cell with alignment pin (facing instrument).

- 13 Make sure that the bottom of the cooling head fully seats on the cell base plate.
- 14 Obtain a long 5/32-inch hexagonal (Allen) wrench from the accessory kit.
- 15 Insert the tip of the wrench into any one of the three captive screws in the LN2P plate while holding onto the cooling head. You may need to push down while you tighten the screw 3 to 4 turns. DO NOT fully tighten yet.



**Figure 14** Installing the LN2P cooling head.

- 16 Repeat step 15 for the two remaining captive screws. After you have started each screw, go back and tighten down all three screws (one or two turns at a time per screw) until you feel them touch the bottom. Do not over tighten.
- 17 Connect the LN2P anti-condensate heater cable, which is attached to the LN2P cooling head, to the +24 VDC port to the left of the DSC cell. See Figure 15.



Figure 15 Installing the heater cable on the DSC.

18 Obtain the DSC top cover and uninstall the plug by removing the two screws holding it in place, as shown below.



Figure 16 Removing plug from DSC top cover.

- 19 Once the cooling head is secured to the cell and the heater cable is properly installed, replace the front cover on the instrument.
- 20 Slide the top cover over the instrument and LN2P hose.

21 Replace the DSC top cover screws that were originally removed, the Autosampler tray, and the Autosampler sample waste bin.

WARNING: Do not place your hands in the path of the Autosampler when it is in motion. Physical injury may occur.

AVERTISSEMENT: Ne placez pas vos doigts sur le chemin de l'échantillonneur automatique lorsqu'il est en mouvement. Des blessures physiques peuvent se produire.

- 22 Select the correct cooler type on the **Discovery DSC** > **Cooler** setting within the TRIOS Software **Options** window.
- 23 Connect the USB cable from the USB port on the LN2P to the USB A port on the Discovery DSC.



Figure 17 Connection on rear of Discovery DSC.

24 Plug the power cord into the power adapter, and then the power adapter into a power outlet. Plug the 24V cable into the 24 VDC 2.5A connector on the LN2P.

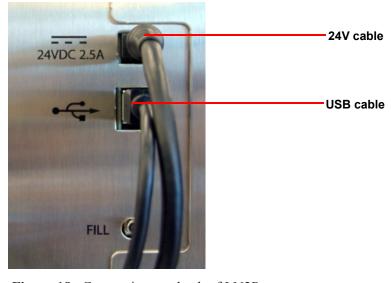


Figure 18 Connections on back of LN2P.

25 Using a Phillips-head screwdriver, remove the strain relief clip identified below. Route the 24V and USB cables through the strain relief clip and replace the clip, as shown below.

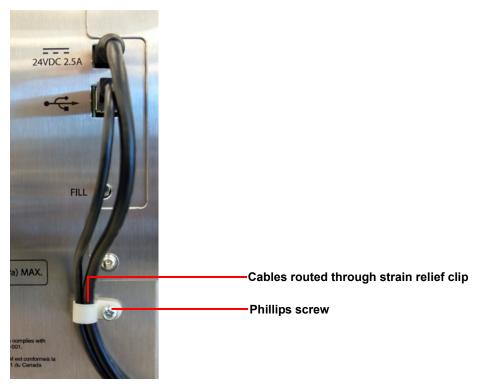


Figure 19 Routing the cables through the strain relief clip.

- 26 Check the AutoLid alignment and adjust, if needed. See TRIOS software Online Help topic "Calibrating the AutoLid" for more information.
- 27 Connect the gas/purge lines as directed in the next section.

## **Connecting Gas Lines**

The **GAS 1** port on the Discovery DSC provides a constant base purge and controlled cell purge; the **GAS 2** port is for cell purge only and can be used with helium to purge the cell. The cooling head secondary purge is connected through the **COOLING GAS** connection. By default, there is no purging of the interior of the LN2P cooling head; however, an option exists in TRIOS Software to automatically purge the interior of the LN2P cooling head when the cell is open during loading/unloading of samples (while under DSC Autosampler control) and during cell conditioning. Refer to TRIOS Help for more information.

Refer to Figure 20 for a diagram of the LN2P gas configuration.

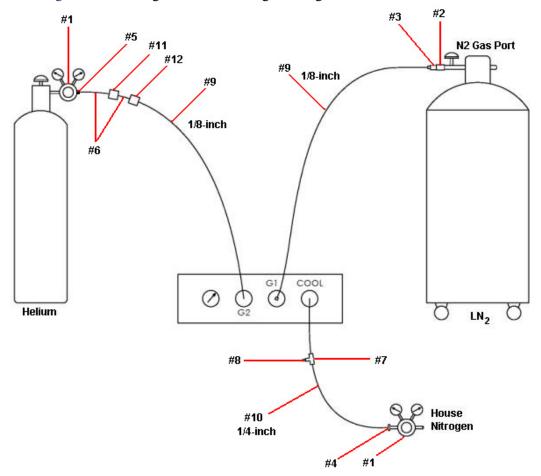


Figure 20 LN2P gas plumbing diagram.

Item	Quantity	Description	Part Number
1	2	Regulator, Concoa #212-2301-01-580	200245.001
2	1	Adapter 1/2 Tube x 1/2 AN	280350.001
3	1	Reduce Union Tube Fitting 1/2 x 1/8-inch Tube OD	200140.004
4	1	LN2 Supply Line	991441.001
5	1	Legris #3175-56-14 1/4 OD Tube to 1/4 Male NPT	270141.002
6	1	Legris #3175-53-14 1/8 OD Tube to 1/4 Male NPT	270141.004
7	1	Legris Plug-in Reducer 1/8 OD x 1/4 OD	200176.001
8	1	Legris #3104-56-53 Tee 1/4 x 1/8 Tube	271648.001
9	1	Legris #3126-53-00 Plug 1/8 OD	271647.002
10	15 ft	Tubing NEXPOLY FR LLDPE 1/8-inch OD	200864.001
11	25 ft	Tubing POLYFLAME 1/4-inch OD	200866.001
12	1	Gas Dryer	200266.001
13	1	Purge Gas Purifier	970425.901

#### Notes:

- Items 1, 12, and 13 are available as parts on the Item Master Price List
- Items 2 4 & 9 are in the LN2P Accessory Kit (972398.901)
- Items 5 8, 10 & 11 are in the Discovery DSC Accessory Kit (972012.901)
- Remove 1/8 tubing from G1 & Tee, then plug with item 9 before attaching item 4.
- Use new or recently serviced and calibrated regulators.
- Do not use Tygon due to its high moisture permeability.
- Make sure that the tubing is cut cleanly and squarely on the ends. Use of the Legris Tubing Cutter #3000-71-00 is recommended.
- Leak check all tubing.
- Use 99.999% pure Helium to reduce moisture buildup in the cell.
- Use the gas dryer, PN 200266.001, to pre-dry and indicate unsatisfactory moisture levels.
- Use the purge gas purifier, PN 970425.901, to achieve a dewpoint of -180°C.

Follow the instructions below to connect the purge lines.

1 Locate the gas ports on the back of the DSC.

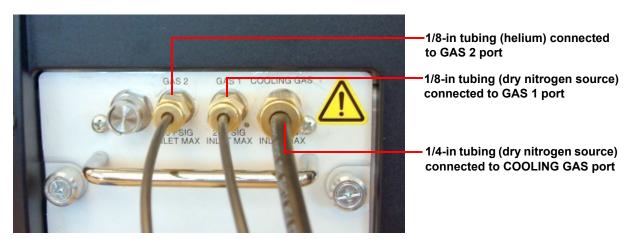


Figure 21 Gas ports on back of Discovery DSC.

2 Ensure that the pressure of your cooling gas is regulated to 140 kPa gauge (20 psig). Dry nitrogen should be used.

**NOTE**: The gases used should be moisture-free. Nitrogen gas of high purity is recommended and can be obtained from a liquid nitrogen source as vaporized gas rather than a compressed gas cylinder.

- 3 Use 1/4-inch O.D. tubing to connect the dry nitrogen gas source to the **COOLING GAS** port on the rear of the DSC. Teflon<sup>®</sup> TFE tubing with Swagelok fittings is recommended. A solenoid valve is automatically controlled by the instrument and can be turned on or off from within TRIOS Software, while the flow rate is limited by an orifice inside the instrument.
- 4 Use 1/8 inch OD tubing to connect the dry nitrogen gas source to the **GAS 1** port. Again, ensure that the pressure of your cooling gas is regulated to 140 kPa gauge (20 psig). The base purge flow rate is determined by an internal orifice and the line pressure. Adjust the line pressure to achieve approximately 300 mL/min flow rate while making sure not to exceed the 140 kPa gauge (20 psig) pressure limit.
- 5 Use 1/8-inch O.D. tubing to connect the helium gas source to the **GAS 2** port. Teflon TFE tubing with Swagelok fitting is recommended. Set the pressure to 103 kPa (15 psig); in TRIOS Software, set the flow rate to 25 mL/min.



**Figure 22** Gas line configuration for DSC-LN2P system.

6 Refer to Chapter 3 for information regarding filling, using, and maintaining the LN2P.

# Chapter 3:

## Operating and Maintaining the LN2P

The LN2P must be filled from a bulk storage tank of liquid nitrogen. There are two methods that can be used to fill the LN2P:

- *Autofilling* refers to the automatic filling of the LN2P from any source. Local autofilling is the most convenient method of filling the tank. Autofill uses a function controlled from the Discovery DSC instrument control software (TRIOS software).
- *Remote filling* is the method that must be used when filling the tank at a location away from the Discovery DSC instrument. Electricity is still required for remote filling. Remote fill uses a program in the LN2P to automatically fill the tank to the proper level.

This chapter discusses the different methods used to fill the LN2P, along with information on conditioning, using, and maintaining the LN2P.

## Connecting and Autofilling the LN2P

CAUTION: Do not use other lines to fill the LN2P, as lower flow resistance will increase Dewar pressure.

MISE EN GARDE: N'utilisez pas d'autres conduites pour remplir le LN2P, car une faible résistance à l'écoulement augmente la pression du Dewar.

To use the local filling capability, connect the cooling accessory to a low pressure bulk source of liquid nitrogen, then fill the Dewar.

There are several ways to autofill the Dewar:

• Using the Common Cabinet User Interface, if available. Select **LN2P Controls** from the **Set View** options list, then press **Fill Tank**.



Figure 23 LN2P Controls screen on User Interface.

- Using TRIOS software:
  - Within the **DSC Cooler** options, enable the autofill function and set a % level; the Dewar automatically fills whenever the liquid nitrogen level reaches the user-set % level.
  - Within the procedure, add one of two **Fill Cooler** segments. One Fill segment waits for the fill process to complete before moving on to the next segment, while the other initiates a fill and moves on to the next segment while still filling the Dewar.
  - Within the General Control panel, click on the Toggle Fill icon to fill the Dewar at any time.

Refer to TRIOS software Online Help for more information on autofilling the Dewar.

## Remote Filling of the LN2P

WARNING: When performing the remote fill procedure described on page 33, always ensure that the manual cap fittings are securely in place on the supply and return lines BEFORE filling the LN2P. If the supply and return lines are not capped, they will discharge liquid nitrogen during remote filling.

AVERTISSEMENT: Lorsque vous effectuez la procédure de remplissage à distance décrite en page 33, assurez-vous toujours que les raccords manuels de capuchon sont solidement installés sur les conduites d'alimentation et de retour AVANT de remplir le LN2P. Si les conduites d'alimentation et de retour ne sont pas fermées, elles vont libérer de l'azote liquide pendant le remplissage à distance.

In addition to autofill, there is the ability to perform a remote fill sequence at a remote location (away from the controller and instrument), if power is available to run the unit. Pressing and holding the **Fill** button on the back of the motor housing for approximately 5 seconds initiates (and terminates) a remote fill.

**NOTE**: Power must be available at the remote filling location for remote fill to function.

Follow the directions in this section to fill the LN2P automatically at a remote location:

- 1 Unplug the power cord, but leave the 24 volt power cord connected to the LN2P, as the power adapter is required at the remote fill location.
- 2 Disconnect the USB cable from the LN2P.

**NOTE**: TA Instruments recommends that you allow the flange on the DSC to reach room temperature before removing any covers or disconnecting the transfer line; doing so ensures no liquid is left in the delivery line.

3 Loosen the two thumb screws on either side of the front motor housing. Lift the cover up and away from the LN2P to remove it.



Thumbscrew to remove (thumbscrew on opposite side not shown)

Figure 24 Thumbscrew on motor housing.

4 Remove the Velcro strap from around the insulation block.



Figure 25 Velcro on insulation block.

- 5 Remove the manual fill cap fittings from the insulation block and allow them to hang from their tethers.
- 6 Carefully open the insulation block while removing it from the LN2P. This exposes the two connectors that will be disconnected from the feed hose.



Figure 26

- 7 Using an 11/16-inch wrench, remove the larger return line from its fitting.
- **8** Using a 9/16-inch wrench, remove the smaller liquid delivery line from its fitting.
- **9** Pull the feed hose off the LN2P.

10 Install the manual fill cap fittings onto the supply and return lines, as shown below.

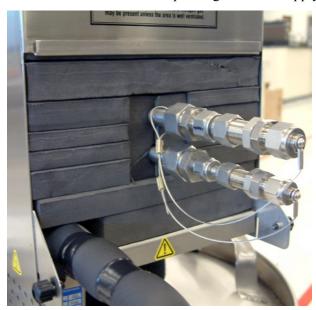


Figure 27 Manual fill cap installed on supply and return lines.

WARNING: When performing the remote fill procedure, always ensure that the manual cap fittings are securely in place on the supply and return lines BEFORE filling the LN2P. If the supply and return lines are not capped, they will discharge liquid nitrogen during remote filling.

AVERTISSEMENT: Lorsque vous effectuez la procédure de remplissage à distance, assurezvous toujours que les raccords manuels de capuchon sont solidement installés sur les conduites d'alimentation et de retour AVANT de remplir le LN2P. Si les conduites d'alimentation et de retour ne sont pas fermées, elles vont libérer de l'azote liquide pendant le remplissage à distance.

- 11 Roll the LN2P to the location of the bulk storage source and plug the power cord into the closest power outlet.
- 12 Make sure that the bulk storage source that will be used for filling the LN2P is a low pressure (maximum 25 psi) container.

13 Connect the transfer hose from the bulk source to the autofill fitting shown below.

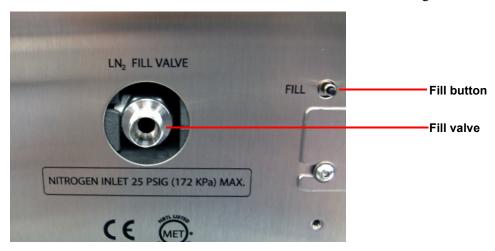


Figure 28 LN2P fill valve.

- 14 Open the valve on the bulk storage source.
- 15 Press and hold the **Fill** button (shown in the figure above) on the LN2P motor housing for 5 seconds to initiate the autofill. The filling will stop automatically when the Dewar is full.

Cold gas will escape from the LN2P vent during the filling process. The fill process normally takes 15 to 40 minutes, depending on the liquid level.

**NOTE**: To stop the fill process, press and hold the **Fill** button for 5 seconds. The LN2P will continue to expel gas from the vent for an additional 2 minutes.

**NOTE**: Frost will build up on the tubing and parts of the LN2P and storage tank while the liquid nitrogen is being transferred.

- 16 After the autofill has completed, allow sufficient time for any liquid remaining in the transfer tube to vaporize. Be aware that the LN2P will continue to expel gas from the vent for an additional 2 minutes after fill is complete.
- 17 Close the valve on the nitrogen bulk storage tank and disconnect the transfer tube from the LN2 Fill Valve.
- **18** Unplug the power cord.
- 19 Return the LN2P to its location near the analysis instrument, and reconnect the cooling accessory by reversing step 1 to step 9.

## Using the LN2P

Once the LN2P has been properly installed, follow the steps below to set up the instrument parameters and start an experiment.

## **Configuring the LN2P**

To configure the LN2P in TRIOS software, see "Configuring the LN2P" Help topic in TRIOS software Online Help.

## **Starting an Experiment**

Before you start the experiment, ensure that the DSC is connected with the controller, the **COOLING GAS**, **GAS** 1, and **GAS** 2 connections are made, and that you have entered all necessary information through TRIOS Software.

**NOTE**: Once the experiment is started, operations are best performed at the computer keyboard or the Discovery DSC user interface display. The DSC is very sensitive to motion and might pick up the vibration caused by touching a key on the instrument keypad.

Start the experiment by selecting **Start** in TRIOS software or by touching the **START** key on the Common Cabinet user interface. When you start the instrument, the system automatically runs the experiment to completion.

**NOTE**: The user can begin the cooling (from TRIOS software or the User Interface) in advance to ensure that the flange is cold before executing the test. In any event, if the cooling is off, it starts automatically at the start of a method (if **LN2P** is selected as the **Cooler Type** in TRIOS software).

## Maintaining the LN2P

This section discusses general LN2P maintenance procedures.

If there are problems with the LN2P that this manual does not address, contact TA Instruments for service.

CAUTION: Do not use any cleaning or decontamination method other than what is recommended in this manual.

MISE EN GARDE: N'utilisez aucune méthode de nettoyage ou de décontamination autre que celle recommandée dans le présent manuel.

## Removing the LN2P Cooling Head

Removal of the LN2P cooling head is performed in the reverse order of installation (refer to "Mounting the Cooling Head" on page 22). However, to avoid potentially damaging the graphite gasket located within the cooling head, always ensure that the Discovery DSC flange is at room temperature before attempting to remove the cooling head.

Should you need to replace the LN2P cooling head gasket, refer to <u>"Replacing the LN2P Graphite Gasket"</u> on page 39.

## Cleaning the LN2P

You can clean the LN2P 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: Ne pas utiliser de produits chimiques, de nettoyants abrasifs, de laine d'acier ou des matériaux bruts pour nettoyer l'appareil.

## Replacing the LN2P Fuse

The LN2P contains an internal fuse that is not user-serviceable. If the internal fuse blows, a hazard may exist. DO NOT attempt to replace this fuse yourself. Call your TA Instruments service representative for service.

## **Replacing the LN2P Graphite Gasket**

The LN2P cooling head contains a graphite gasket that acts as a thermal interface between the copper heat exchanger on the cooling head and the nickel flange on the Discovery DSC cell.

The gasket should be replaced if any damage to the gasket is visible. To replace the gasket in the LN2P cooling head:

1 Locate the gasket within the cooling head, as shown below.

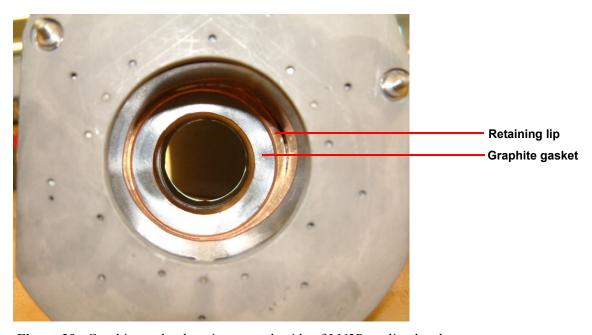


Figure 29 Graphite gasket location on underside of LN2P cooling head.

- 2 Using your fingers, remove the old gasket by sliding it out from under the retaining lip (shown in the figure above) and pulling it out of the cooling head.
- 3 Carefully insert the new gasket into the cooling head, making sure to slide the gasket beneath the retaining lip.
- 4 To ensure proper installation, ensure that the gasket rotates freely beneath the retaining lip. Then turn the LN2P cooling head right side up (gasket facing downwards). The gasket is properly installed if it remains seated within the cooling head.

## **Drying the System**

Under certain conditions, it may be necessary to dry out the DSC-LN2P system. To remove moisture in the DSC cell and cooling head, refer to TRIOS software Online Help topic "Drying the DSC-LN2P System".

## Replacement Parts

The table below lists the replacement parts for the LN2P.

**Table 2: LN2P Replacement Parts** 

Part Number	Description
972410.901	Cooling head assy
202084.001	Power supply
253827.000	Power cord
201258.001	USB cable
972271.901	Gasket, graphite, heat exchanger
972455.001	LN2P tank neck insulation
972457.001	LN2P pump head front insulation
200892.002	Velcro strap neck
200892.003	Velcro strap front