

LIQUID NITROGEN PUMP (LN PUMP)



Getting Started Guide

Revision I

Issued June 2018

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. 61010.1-10 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

For European Economic Area

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

For United States

UL61010-1 2012 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: 2013 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 LNP for your protection:

Symbol	Explanation
\wedge	This symbol on the LNP 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 LNP.
	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 LNP.
	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 LNP 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 LNP 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: 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 LNP 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 (LNP) 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 (LNP) 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 LNP and Discovery DSC system may get extremely cold when using the LNP 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 LNP et du système Discovery DSC peuvent devenir extrêmement froides lors de l'utilisation du LNP 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 38, always ensure that the manual cap fittings are securely in place on the supply and return lines BEFORE filling the LNP. 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 38, assurez-vous toujours que les raccords manuels de capuchon sont solidement installés sur les conduites d'alimentation et de retour AVANT de remplir le LNP. 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 LNP surfaces get cold during use of the LNP. 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 stays dry. A slipping hazard may result if the condensation is not cleaned up.

AVERTISSEMENT: Certaines surfaces du DCS et du LNP deviennent froides pendant l'utilisation du LNP. 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 LNP cooling head installed and the LNP 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 LNP est installée et le LNP n'est pas activé. Cela pourrait provoquer de graves dégâts à la tête de refroidissement.

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

MISE EN GARDE: Nous recommandons de ne pas utiliser le LNP 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 LNP est utilisé à des températures élevées sur de longues périodes.

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Chapter 1:

Introducing the LN Pump

Overview

The Liquid Nitrogen Pump ("LN Pump" or "LNP" in this Guide) is a cooling accessory for use with the TA Instruments Discovery DSC[™]. The LN Pump allows continuous temperature control within the range of −180 to 550°C. The LN Pump delivers (via a pump) liquid nitrogen to the heat exchanger, which in turn cools the cell.

The LN Pump can be controlled from the **General** panel of TRIOS software, the user interface on Discovery DSC 25, DSC 250, and DSC 2500 units, or the Common Cabinet user interface for first generation Discovery DSC units.



Figure 1 LN Pump.

Components

The LN Pump 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 cooling head connection cover. 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 3/8-inch 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 <u>Chapter 3</u>.

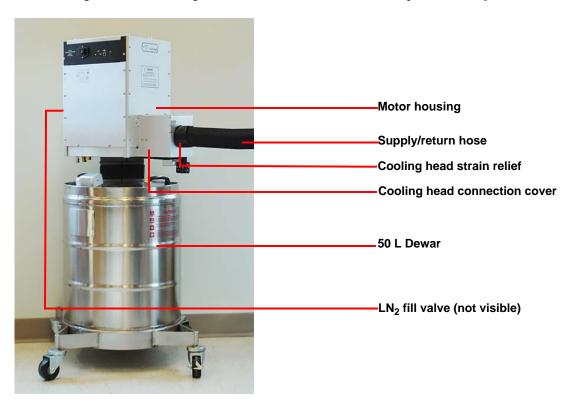


Figure 2 LN Pump components.

Typical setup of the LN Pump and DSC is shown below. Refer to <u>"Positioning the Transfer Line"</u> for more information on requirements for placing the LN Pump in proximity to the instrument and positioning the transfer line.



Figure 3 Typical setup (DSC 2500 shown).

Additionally, a drain tube with condensate catch trough (P/N 991339.902) can be added to the dewar to capture melting ice.

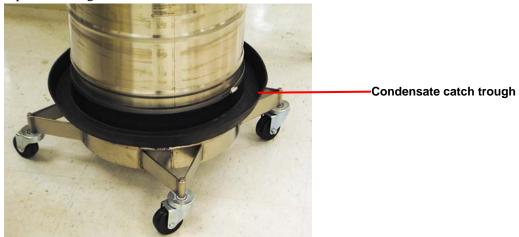


Figure 4 Condensate catch trough (drain tube not shown).

Instrument Specifications

The table found below contains the technical specifications for the LN Pump.

Table 1: LN Pump Technical Specifications

Item/Area	Specification		
Instrument compatibility	Discovery DSC		
Size Height Width Depth	122 cm (48 in) 86 cm (34 in) 86 cm (34 in)		
Weight 50 kg (110 lbs) empty 92.5 kg (204 lbs) full			
Power requirements 100–240 VAC / 2A, 50/60Hz Approved for operation on a 20 amp branch circ tective conductor (ground)			
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	10 kPa gauge (1.5 psig) for pressure regulation		
Liquid nitrogen transfer line	1.8 m (6 ft) insulated from LN Pump to heat exchanger		
Liquid nitrogen fill line	1.8 m (6 ft) insulated from LN Pump to bulk storage. Supplied with union and adapter for bulk storage connection. Do not use other lines to fill the LN Pump, 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 LN Pump when running isothermal experiments above 400°C. The life of the DSC cell heating element can be shortened if the LN Pump is used at high temperatures for extended periods.

MISE EN GARDE: Nous recommandons de ne pas utiliser le LN Pump 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 LN Pump est utilisé à des températures élevées sur de longues périodes.

Chapter 2:

Installing the LN Pump

Unpacking and Inspecting the System

Inspect the contents of the LN Pump shipping box. Check all inner boxes for documentation, parts, and accessories. 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 LN Pump.

If the LN Pump 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 LN Pump

Installation of the LN Pump 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 LN Pump. 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.

CAUTION: If connecting the LN Pump to a Discovery DSC, TRIOS 4.1.1 or higher must be installed on the controller, and the DSC firmware must be updated prior to physically connecting the LN Pump to the DSC via the USB communications cable.

MISE EN GARDE: Si vous connectez la pompe LN à un DSC Discovery, TRIOS 4.1.1 ou supérieur doit être installé sur le contrôleur et le micrologiciel DSC doit être mis à jour avant de connecter physiquement la pompe LN au DSC via le câble de communication USB.

Choosing a Location

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

CAUTION: The order for installation of the LN Pump is:

- 1 Install LN Pump cooling head onto DSC
- 2 Place tank into desired location
- 3 Carefully route transfer line from head to the tank. The supply/return line should approach the DSC cell and the LN Pump straight and parallel from the floor for the maximum cooling efficiency.
- 4 Install the transfer line to the tank

MISE EN GARDE: L'ordre d'installation de la pompe LN est:

- 1 Installer la tête de refroidissement de la pompe LN sur DSC
- 2 Placez le réservoir dans l'emplacement désiré
- 3 Acheminer prudemment la ligne de transfert de la tête vers le réservoir. La de transfert doit s'approcher de la cellule DSC et de la pompe LN droite et parallèle du plancher pour une efficacité de refroidissement maximale.
- 4 Installez la ligne de transfert dans le réservoir

Installing the LN Pump on the Discovery DSC Instruments

Installation of the LN Pump onto the Discovery DSC instrument 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 LN Pump on the Discovery DSC instruments.

Removing the top cover on Discovery DSC 25, DSC 250, DSC 2500

1 If an Autosampler is present, it must be moved away from the cell by pressing the Lid Open/Close

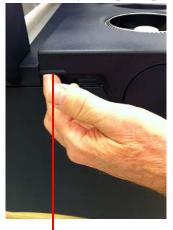


button on the user interface.

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.

2 Carefully lift up and pull the top cover off of the DSC.





Pull up on tab to remove cover

Figure 5 Remove the top cover (DSC 2500 shown).

Removing the top cover on Discovery DSC

1 Press the Lid button on the Discovery DSC keypad to close the AutoLid, as shown in Figure 6.

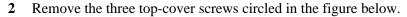




Figure 6 Discovery DSC top cover.

- 3 Press the **Lid** button on the DSC keypad to open the AutoLid on your instrument.
- 4 Remove the Autosampler tray and Autosampler sample waste bin from the top cover. Refer to Figure 6 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.

- 5 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.
- 6 Carefully lift up and pull the top cover away from the Discovery DSC (to the right when facing the instrument).

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.

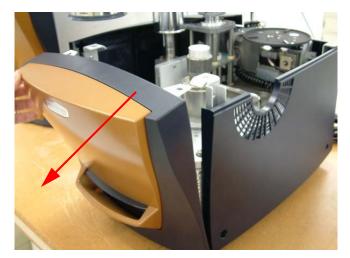


Figure 7 Removing the front cover.

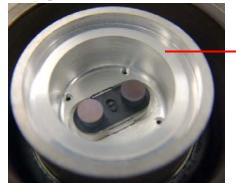
Mounting the Cooling Head

The LN Pump 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 LN Pump unit.

CAUTION: Before beginning, ensure the cell is at a temperature where contact with the cell will not cause a burn.

MISE EN GARDE: Avant de commencer, assurez-vous que la cellule est à une température où le contact avec la cellule ne sera pas provoquer une brûlure.

1 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 on the silver surface indicated in Figure 8, contact your local TA Instruments Service Representative for details on re-dressing the cell.



Verify that this inner cell lip is free of irregularities

Figure 8 Surface of silver block.

2 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 LN Pump 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.

3 For the Discovery DSC 25, DSC 250, DSC 2500: Position the LN Pump in close proximity and to the left of the DSC.

For the Discovery DSC: Position the LN Pump in close proximity and to the right of the DSC.

CAUTION: Take extreme care not to damage the silver block shown in <u>Figure 8</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 13. 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.

4 Align the pin on the cell base (shown in the figure below) with the corresponding slot in the LN Pump 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 9 Exposed DSC cell with alignment pin (facing instrument).

- 5 Make sure that the bottom of the cooling head fully seats on the cell base plate.
- 6 Obtain a long 5/32-inch hexagonal (Allen) wrench from the accessory kit.

Insert the tip of the wrench into any one of the three captive screws in the LN Pump 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.

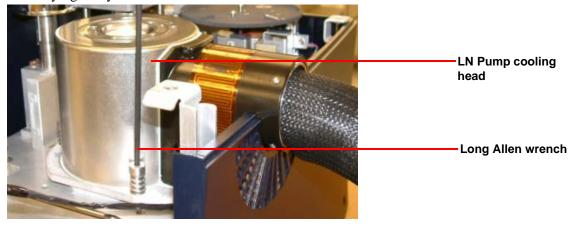


Figure 10 Installing the LN Pump cooling head.

- 8 Repeat step 7 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.
- 9 Connect the LN Pump anti-condensate heater cable, which is attached to the LN Pump cooling head, to the +24 VDC port (for Discovery DSC 25, DSC 250, and DSC 2500, the +24 VDC port is located behind and to the right of the DSC cell; on Discovery DSC, this is to the left of the DSC cell). See Figure 11.



Figure 11 Installing the heater cable on the DSC.

10 Uninstall the plug from the DSC top cover by removing the two screws holding it in place



Figure 12 Removing the plug from DSC top cover (Discovery DSC 2500 shown).

11 For Discovery DSC 25, DSC 250, DSC 2500: Once the cooling head is secured to the cell and the heater cable is properly installed, replace the top cover on the instrument. Make sure the instrument and LN Pump assembly are powered off, and then connect the CAN communication cable to the DSC and the LN Pump connectors labeled "CAN."

For Discovery DSC: Once the cooling head is secured to the cell and the heater cable is properly installed:

- **a** Replace the front cover on the instrument.
- **b** Slide the top cover over the instrument and LN Pump hose.
- **c** Replace the DSC top cover screws that were originally removed, the Autosampler tray, and the Autosampler sample waste bin.
- d Connect the USB communications cable to the DSC and LN Pump connections labeled with a USB icon

CAUTION: If connecting the LN Pump to a Discovery DSC, TRIOS 4.1.1 or higher must be installed on the controller, and the DSC firmware must be updated prior to physically connecting the LN Pump to the DSC via the USB communications cable.

MISE EN GARDE: Si vous connectez la pompe LN à un DSC Discovery, TRIOS 4.1.1 ou supérieur doit être installé sur le contrôleur et le micrologiciel DSC doit être mis à jour avant de connecter physiquement la pompe LN au DSC via le câble de communication USB.

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.

12 Select the correct cooler type on the **Discovery DSC** > **Cooler** setting within the TRIOS Software **Options** window.

- 13 Check the AutoLid alignment and adjust, if needed. See TRIOS software Online Help topic "Calibrating the AutoLid" for more information.
- 14 Plug the instrument end of the power cord into the power entry module on the back of the LN Pump, then plug the mains end of the power cord into a power outlet.



Figure 13 Power entry module.

15 Connect the gas/purge lines as directed in the next section.

Positioning the Transfer Line

The total length of the transfer line from DSC to LN Pump is 152 cm (60 in). The transfer line should approach the DSC cell and the LN Pump straight and parallel from the floor for maximum cooling efficiency. A minimal straight length of 31 cm (12 in) is required. The remaining 122 cm (48 in) is flexible with no hard bends (at an approximate 90 degree angle). After the LN Pump cooling head is fixed to the DSC cell, make any necessary bends to the center of the transfer line to align the pump.



Figure 14 Transfer line position.

Installing the Transfer Line

Before the transfer line is connected to the tank, the cooling head must first be installed on the DSC. Refer to "Mounting the Cooling Head" on page 21. Follow the instructions below to install the transfer line.

1 Using a Phillips screwdriver, loosen the four screws on the top of head connection cover. Then loosen the two thumbscrews on the front of the cover (a Phillips screwdriver can be used, if needed). The cover is removed in two pieces (top: under the four screws, and front: fastened with two thumbscrews). Lift the cover pieces up and away from the LN Pump to remove it.

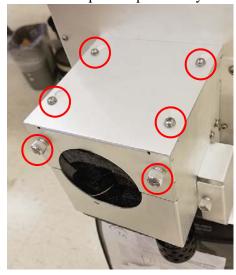


Figure 15 Left: Head connection cover thumbscrews (DSC 25XX).

2 Remove the insulation to expose the two fittings that will be attached to the feed hose.

3 Using a 3/4-inch wrench and a 11/16-inch wrench, remove the return line connection cap to vent the tank. Then, using a 3/4-inch wrench and a 9/16-inch wrench, remove the pressure relief valve.



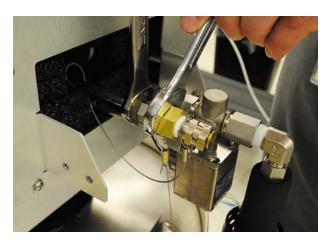


Figure 16 Top: Remove the return line connection cap. Bottom: Remove the pressure relief valve.

4 Obtain the 1.8-m (6 ft) long transfer line with its attached cooling head. At the opposite end from the cooling head are two fittings that need to be connected to the LN Pump tank. The figure here identifies the two fittings that will be attached. Note that the return line fitting is larger than the supply line fitting.

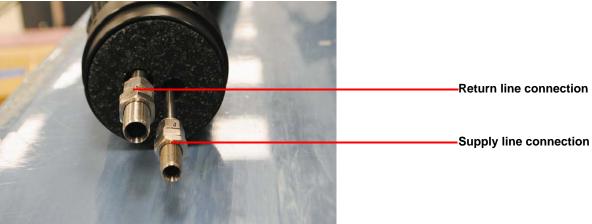


Figure 17 Two fittings to connect to LN Pump tank.

- 5 Connect the transfer line to the LN Pump:
 - a Connect the 9/16 bulkhead fitting to the nut on copper tubing of the coolant head.

NOTE: The bulkhead fitting is not attached during shipping and is packaged separately in a bag attached to the coolant head.



Figure 18 Harness cooling head to instrument (PN 972209.901).

b Using a 9/16-inch wrench, connect the nut to the other end of the bulkhead.

CAUTION: A wrench must be used when attaching the Swagelock nut to prevent the supply line from twisting and being damaged.

MISE EN GARDE: Une clé doit être utilisée lors de la fixation de l'écrou Swagelock pour éviter que la ligne d'alimentation ne se torde et soit endommagée.

c Using a 9/16-inch and a 5/8-inch wrench, attach the supply line connection on the transfer hose to the supply line connection on the LNP (see top image in <u>Figure 19</u>. Using an 11/16-inch and a 3/4-inch wrench, attach the return line on the transfer hose to the remaining fitting on the LNP (see bottom image in <u>Figure 19</u>).

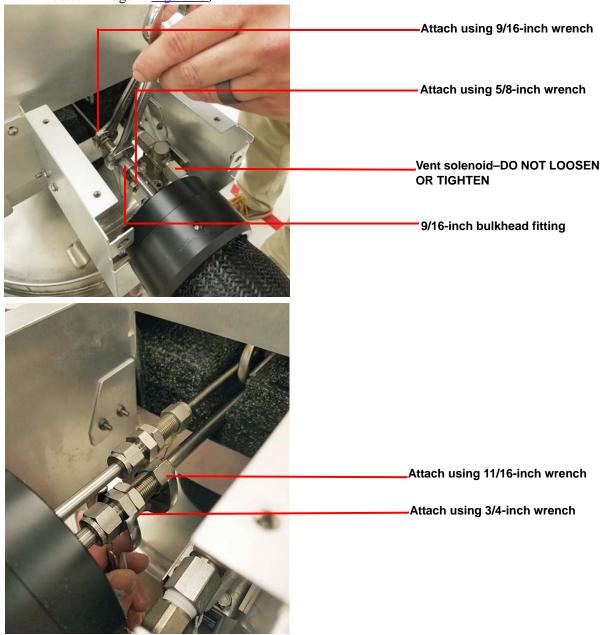


Figure 19 Top: Supply line attached to the transfer hose. Bottom: Return line attached to the transfer hose.

CAUTION: A wrench must be used when attaching the Swagelock nut to prevent the supply line from twisting and being damaged.

MISE EN GARDE: Une clé doit être utilisée lors de la fixation de l'écrou Swagelock pour éviter que la ligne d'alimentation ne se torde et soit endommagée.

6 Carefully replace the insulation block around the fittings:

a Insert insulation piece #1 up through the bottom of the head connection cover.





Figure 20 Insulation piece #1

b Insert insulation piece #2 in the space to the left of the connections in the head connection cover.



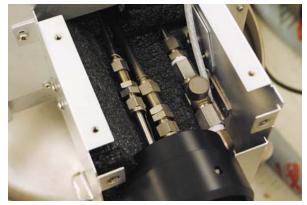


Figure 21 Insulation piece #2

c Insert insulation piece #3 over top of the fittings, with the flat side facing up so that the fittings are completely covered.

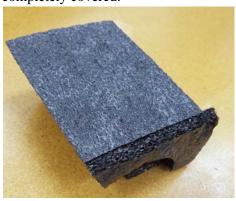




Figure 22 Insulation piece #3

d Slide insulation piece #4 under the head connection cover brackets to cover insulation pieces 2 and 3.



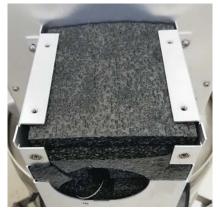


Figure 23 Insulation piece #4

- 7 Replace both of the head connection cover pieces, making sure to tighten the thumbscrews finger tight.
- 8 Slide the strain relief insulation over the transfer line so that it is positioned over the strain relief and against the metal head connection cover. Use the velcro straps to tighten the insulation.



9 With the LN Pump in its final position, lock the two locking casters to prevent movement that could impact operation of the system.

Perform these steps in reverse to uninstall the transfer line.

Installing the LN2 Pump to the Tank

Ice and frost are created during normal use of the LN Pump. 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, make sure that the transfer line and relief valves are not aligned above the handles or vacuum seal cap.

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, <u>Figure 24</u> 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 <u>Figure 24</u> montre l'orientation de la pompe vers le réservoir.



Figure 24 Pump and tank orientation.

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 LN Pump cooling head; however, an option exists in TRIOS software to automatically purge the interior of the LN Pump cooling head when the cell is open during loading/unloading of samples (while under DSC Autosampler control) and during cell conditioning. Refer to TRIOS Online Help for more information.

Refer to "Appendix: Plumbing Diagrams" for diagrams of the DSC instrument gas configuration when using the LN Pump.

Follow the instructions below to connect the purge lines.

- 1 Locate the gas ports on the back of the DSC:
 - a Discovery DSC 25, DSC 250, DSC 2500 gas ports:



Figure 25 Gas ports on back of DSC 25, 250, 2500.

b Discovery DSC gas ports:



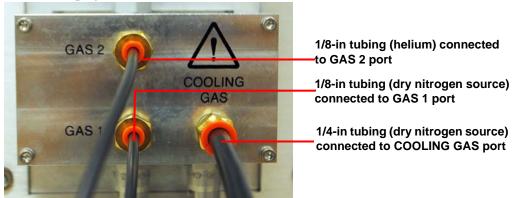
Figure 26 Gas ports on back of Discovery DSC.

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. 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. Set the pressure to 103 kPa (15 psig); in TRIOS software, set the flow rate to 25 mL/min.



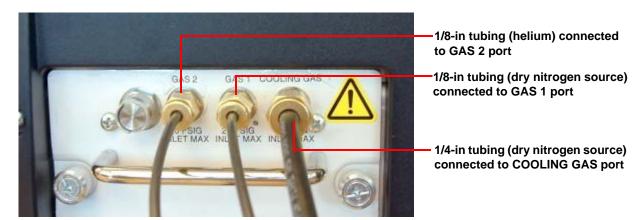


Figure 27 Gas line configuration for DSC-LN Pump system: Discovery DSC 25, DSC 250, DSC 2500 (top); Discovery DSC (bottom).

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

Chapter 3:

Operating and Maintaining the LN Pump

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

- Autofilling refers to the automatic filling of the LN Pump 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 LN Pump to automatically fill the tank to the proper level.

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

Connecting and Autofilling the LN Pump

WARNING: Do not use other lines to fill the LN Pump, as lower flow resistance will increase Dewar pressure.

AVERTISSEMENT: N'utilisez pas d'autres conduites pour remplir le LN Pump, 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:

• Discovery DSC 25, DSC 250, DSC 2500 user interface: Press **Settings**, and then set LNP AutoFill to **On**.

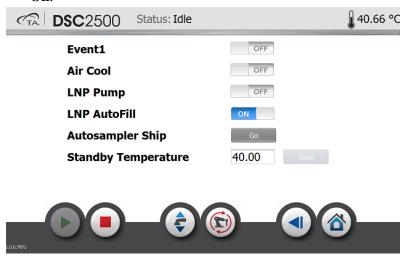


Figure 28 LNP AutoFill set to On.

• Discovery DSC Common Cabinet user interface (if available): Select **LN2P Controls** from the **Set View** options list, then press **Fill Tank**.



Figure 29 LNP 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 LN Pump

WARNING: When performing the remote fill procedure described on page 38, always ensure that the manual cap fittings are securely in place on the supply and return lines BEFORE filling the LN Pump. 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 38, assurez-vous toujours que les raccords manuels de capuchon sont solidement installés sur les conduites d'alimentation et de retour AVANT de remplir le LN Pump. 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 LN Pump automatically at a remote location:

1 Make sure the instrument and LN Pump assembly are powered off, then disconnect the CAN or USB cable from the LN Pump. Disconnect the power cord from the mains outlet.

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.

- 2 Uninstall the transfer line. Refer to <u>"Installing the LN2 Pump to the Tank" on page 32</u>, performing the procedure in reverse order to uninstall the transfer line. Make sure to remove the return line first to vent the tank prior to removing the liquid delivery line.
- 3 Screw on the caps over the liquid delivery 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 LN Pump. 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 LN Pump. Si les conduites d'alimentation et de retour ne sont pas fermées, elles vont libérer de l'azote liquide pendant le remplissage à distance.

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

6 Connect the fill hose from the bulk source to the autofill fitting shown below.



Figure 30 LN Pump fill valve.

- 7 Open the valve on the bulk storage source.
- 8 Press and hold the **Fill** button on the LN Pump motor housing for 5 seconds to initiate the autofill. The filling will stop automatically when the Dewar is full.



Figure 31 LN Pump fill button.

Cold gas will escape from the LN Pump 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 LN Pump 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 LN Pump and storage tank while the liquid nitrogen is being transferred.

- 9 After the autofill has completed, allow sufficient time for any liquid remaining in the fill tube to vaporize. Be aware that the LN Pump will continue to expel gas from the vent for an additional 2 minutes after fill is complete.
- 10 Close the valve on the nitrogen bulk storage tank and immediately disconnect the fill tube from the LN2 Fill Valve.
- 11 Unplug the power cord.

12 Return the LN Pump to its location near the analysis instrument. Remove the return fitting first and allow the Dewar to vent before removing the supply cap. Reconnect the cooling accessory by reversing step 1 to step 10.

Using the LN Pump

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

Configuring the LN Pump

To configure the LN Pump in TRIOS software, see "Configuring the Instrument" Help topic in TRIOS software Online Help.

Starting an Experiment

CAUTION: TRIOS 4.1.1 or higher must be installed on the controller before starting experiments or upgrading firmware

MISE EN GARDE: TRIOS 4.1.1 ou supérieur doit être installé sur le contrôleur avant de démarrer une expérimentation ou une mise à niveau du micrologiciel.

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 or the DSC 25, DSC 250, DSC 2500 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 **LN Pump** is selected as the **Cooler Type** in TRIOS software).

Maintaining the LN Pump

This section discusses general LN Pump maintenance procedures.

If there are problems with the LN Pump 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 LN Pump Cooling Head

Removal of the LN Pump cooling head is performed in the reverse order of installation (refer to "Mounting the Cooling Head" on page 21). 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 LN Pump cooling head gasket, refer to <u>"Replacing the LN Pump Graphite Gasket" on page 43.</u>

Cleaning the LN Pump

You can clean the LN Pump 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 LN Pump Fuse

The LN Pump also 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.

The LN2 power entry module contains two fuses. Replace only with the same type and rating as indicated on the panel adjacent to the power entry module.

- 1 Turn power off on the LN Pump and remove the power cord.
- 2 The power entry module has one standard fuse holder built in that holds two fuses. Use a flat-blade screwdriver to remove the fuse carrier.

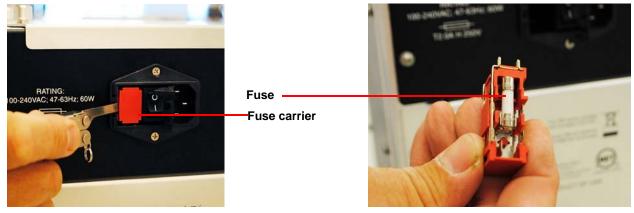


Figure 32 Removing the fuse carrier and fuses.

- **3** Remove old fuses and replace the fuses only with the type and rating indicated on the panel adjacent to the power entry module.
- 4 Place the fuse carrier back into the fuse holder and snap the cover in place.

Replacing the LN Pump Graphite Gasket

The LN Pump 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 LN Pump cooling head:

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

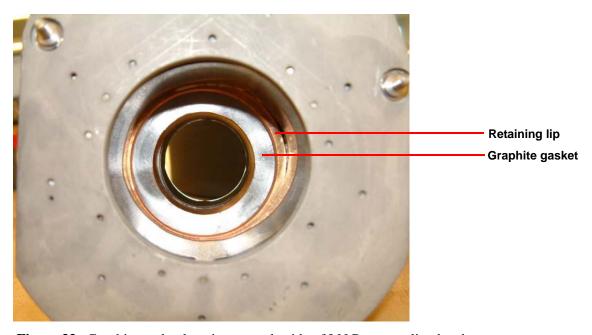


Figure 33 Graphite gasket location on underside of LN Pump cooling head.

- 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 LN Pump 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-LN Pump system. To remove moisture in the DSC cell and cooling head, refer to TRIOS software Online Help topic "**Drying the DSC-LN Pump System**".

Replacement Parts

The table below lists the replacement parts for the LN Pump.

Table 2: LN Pump Replacement Parts

Part Number	Description
973899.901	Cooling Head Assembly
200121.002	Valve Relief 50 psi
200121.001	Valve Pressure Safety Relief 13 psi
200121.005	Valve Regulator 1.5 psi
973973.001	Insulation Strain Relief
200892.004	Velcro strap
973987.901	LN Pump Head Neck Insulation
973710.901	Kit Insulation Transition LNP
973990.901	Harness Communication LNP
201242.005	Fuse Type T 250 V 2 A
253827.000	Power Cord 14AWG 3 COND
991441.001	Line Fill N2 Supply GCA
972271.901	Gasket Heat Exchanger CCS
201258.001	USB cable
973990.901	CAN communications cable
973902.901	Pump Assembly

Appendix:

Plumbing Diagrams

Diagram 1: Typical Gas Connections

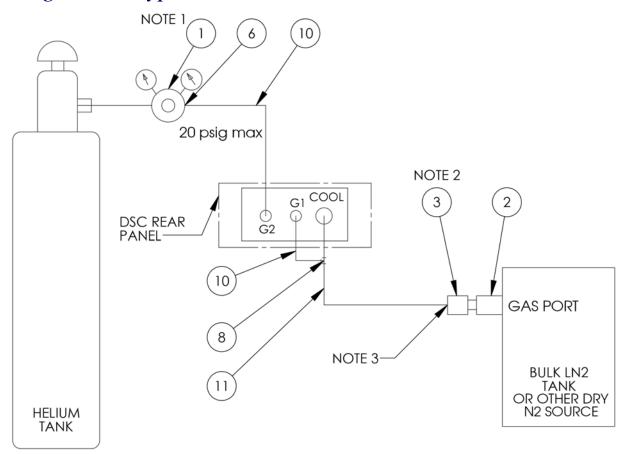


Figure 34

Diagram 2: With Optional Gas Dryer

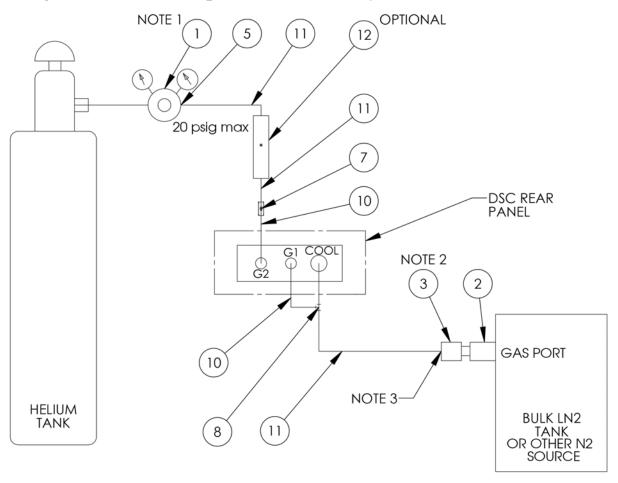


Figure 35

Diagram 3: With Optional Gas Dryer, Purge Gas Purifier, and Bypass Manifold

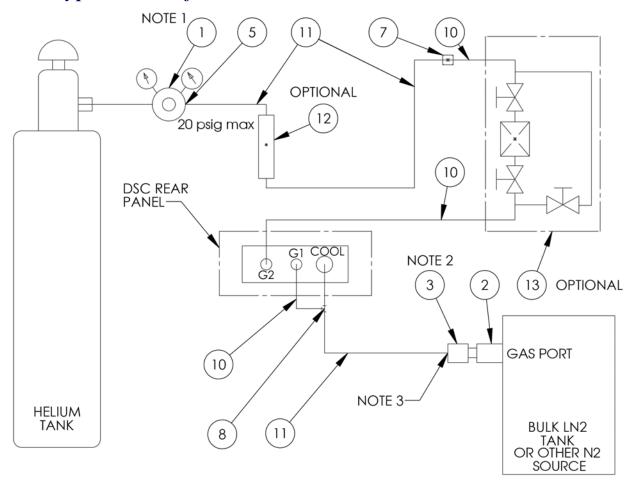


Figure 36

NOTES:

- 1 Set regulator (Item 1) downstream pressure to 20 psig max, 5 psig recommended.
- 2 Remove and discard 1/2-inch nut and ferrule from Item 3 before connecting it to Item 2.
- 3 Dry nitrogen gas connection 20 psig max. LN2 boil-off recommended.
- 4 Items 1, 12, and 13 are available for purchase, contact your TA sales representative.
- 5 Items 2 & 3 are in the LN Pump Accessory Kit (973911.901)
- 6 Items 5 8, 10 & 11 are in the Discovery DSC Accessory Kit (973012.901, 973012.902, and 973012.903)
- 7 Use new or recently serviced and calibrated regulators.
- **8** Do not use Tygon due to its high moisture permeability.
- 9 Make sure that the tubing is cut cleanly and squarely on the ends. Use of the Legris Tubing Cutter #3000-71-00 is recommended.
- 10 Leak check all tubing.
- 11 Use 99.999% pure Helium to reduce moisture buildup in the cell.
- 12 Use the gas dryer (Item 12), PN 200266.001, to pre-dry and indicate unsatisfactory moisture levels. See "Diagram 2: With Optional Gas Dryer" or "Diagram 3: With Optional Gas Dryer, Purge Gas Purifier, and Bypass Manifold".
- 13 Use the purge gas purifier (Item 13), PN 970425.901, to achieve a dewpoint of –180°C. See "Diagram 3: With Optional Gas Dryer, Purge Gas Purifier, and Bypass Manifold".

Item	Quantity	Description	Part Number
1	1	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 1/4-inch Tube OD	200140.005
4	1	LN2 Fill Line (see "Remote Filling of the LN Pump" on page 38)	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 #3106-53-56 Union 1/8 to 1/4 Tube	200362.001
8	1	Legris #3104-56-53 Tee 1/4 x 1/8 Tube	271648.001
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 & Bypass Manifold	970425.901