LNCS
Liquid Nitrogen Cooling System

Q Series™
Getting Started Guide

Revision G
Issued January 2004

109 Lukens Drive
New Castle, DE 19720

Notice

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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

A NOTE highlights important information about equipment or procedures.

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

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Regulatory Compliance

Safety Standards

For Canada:

CAN/CSA-22.2 No. 1010.1-92 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.
CAN/CSA-22.2 No. 1010.2.010-94 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For the European Economic Area: (In accordance with Council Directive 73/23/EEC of 19 February 1973 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN61010-1: 1993 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.
EN61010-2-010: 1994 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For the United States:

UL3101-1 Electrical Equipment for Laboratory Use; Part 1: General Requirements.
IEC 1010-2-010: 1992 Particular requirements for laboratory equipment for the heating of materials + Amendments.

Electromagnetic Compatibility Standards

For Australia and New Zealand:

AS/NZS 2064: 1997 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radiofrequency equipment.

For Canada:

ICES-001 Issue 3 March 7, 1998 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area: (In accordance with Council Directive 89/336/EEC of 3 May 1989 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1: 1997 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements + Amendments (for 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 radiofrequency emissions).

Safety

CAUTION: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the safety protection designed into the equipment may be impaired.

CAUTION: Due to the size and weight of the cooling accessory, the LNCS should always be lifted by two people to prevent injury.

CAUTION: 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.

Electrical Safety

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 Vac are present in this system.

WARNING: High voltages are present in this instrument. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

Handling Liquid Nitrogen

The LNCS 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.

1. Wear goggles or a face shield, gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.

2. Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.

3. The purity of liquid nitrogen decreases when exposed to air. If the liquid in a container has been open to the atmosphere for a prolonged period, analyze the remaining liquid before using it for any purpose where high oxygen content could be dangerous.

WARNING:
Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent the Liquid Nitrogen Cooling System (LNCS) container in confined spaces.

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

The warning on this page applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use.

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 DSC, cold could also cause injury. After running any type of experiment, you must allow the DSC cell to return to room temperature before you touch the inner cell surfaces.

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

Water Condensation

WARNING: Some of the DSC and LNCS surfaces get cold during use of the LNCS. The cold surfaces can cause condensation and, in some cases, frost to build up. This condensation may drip to the floor. Provisions to keep the floor dry should be made. A slipping hazard may result if the condensation is not cleaned up.

Temperature Range

CAUTION: Do not exceed 100°C with the LNCS cooling head installed and the LNCS power off. Serious damage to the cooling head could occur.

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

Chapter 1
Introducing the LNCS

Overview

The LNCS (Liquid Nitrogen Cooling System) is a cooling accessory for use with TA Instruments Analyzers. It can be used with the Differential Scanning Calorimeter (DSC) models Q100 and Q1000.

The Liquid Nitrogen Cooling System (LNCS) allows automatic and continuous temperature control within the range of –180°C to 550°C. The LNCS tank is pressurized to deliver the liquid nitrogen to the heat exchanger, which in turn cools the cell.

The LNCS (shown here) can be autofilled in your laboratory using the DSC touch screen or through the instrument control software. This requires a low pressure 170 kPa gauge maximum (25 psig) bulk storage tank to be located within 1.8 m (6 feet) of the LNCS. See Chapter 3 for information on filling.

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

Specifications

The specifications in Table 1 apply to the Liquid Nitrogen Cooling System.

Table 1
Technical Specifications

LNCS liquid nitrogen capacity	50 L
Size	115 cm (45 in.) high by 48 cm (19 in.) in diameter
Power requirements	100–240 Vac; 47–63 Hz, 180 VA
Weight	51 kg (113 lbs) empty; 87 kg (193 lbs) full
Cooling capacity	–180°C
Pressure relief	90 kPa gauge (13 psig) for Dewar 345 kPa gauge (50 psig) for fill line
Pressure gauge	0 to 210 kPa gauge (0 to 30 psig)
Liquid nitrogen feed hose	1.8 m (6 ft) insulated from LNCS to heat exchanger.
Liquid nitrogen fill hose	1.8 m (6 ft) insulated from LNCS to bulk storage. Supplied with union and adapter for bulk storage connection.
Bulk storage tank	Use low pressure supply tank only. Recommended source pressure is 140 to 170 kPa gauge (20 to 25 psig).

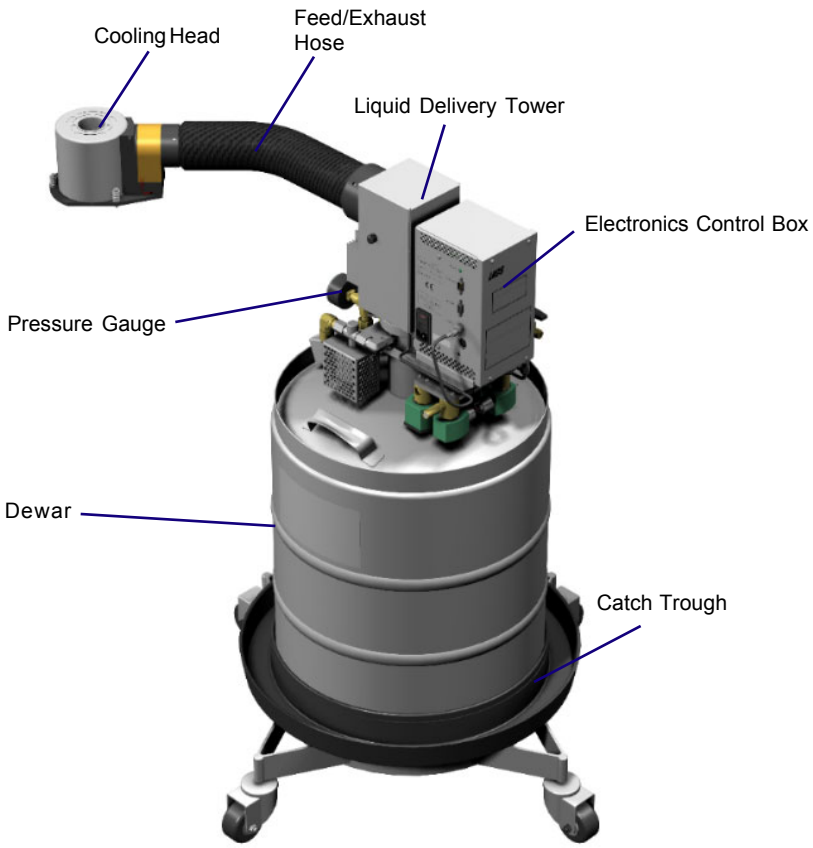
Components

The LNCS is made up of a 50-liter dewar, liquid delivery tower, an electronics control box, and a cooling head that is connected to the control box by a 1.8-meter (6-foot) long feed/exhaust hose. See the figure below.

The dewar can be pressurized either internally or externally. *Internal pressurization* limits the capabilities (primarily temperature responsiveness) of the cooler as the liquid level decreases. It also consumes more liquid nitrogen, therefore reducing the available usage time of the dewar.

External pressurization is preferred for optimal performance and is accomplished with a nitrogen gas source at the users facility regulated to between 55 and 70 kPa gauge (8 and 10 psig). A three-way valve on the LNCS controls the operating mode.

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



Major Components of the LNCS

- The first two are for connection of the cooling head and feed/exhaust hose. They are located at the top of the liquid delivery tower. One fitting (0.25-inch tube) is for the LN2 supply to the heat exchanger (in the cooling head) and the other fitting (3/8-inch tube) is for the exhaust gas from the heat exchanger. For instructions on attaching these lines, see Chapter 2: "Installing the LNCS."
- There are two fittings for connection to a bulk LN2 source for filling purposes, one which is controlled by a solenoid valve and the other for manual filling. For instructions on the use of this port see Chapter 3: "Filling, Use, Conditioning, & Maintenance."
- The last fitting is the attachment point for the external dewar pressurization line, an 55 to 70 kPa gauge (8 to 10 psig) nitrogen source, which is used to control the operating pressure of the dewar to deliver nitrogen to the heat exchanger. Use of this port is optional as self-pressurization is available.

Chapter 2
Installing the LNCS

Unpacking and Inspecting

By the time you are reading this manual, you have already done a certain amount of unpacking. Continue to unpack and inspect the contents of the LNCS 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 LNCS.

If the LNCS 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 LNCS

Installation of the LNCS 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 LNCS. Whenever you handle liquid nitrogen, wear goggles or a face shield and gloves large enough to be removed easily.

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

Choosing a Location

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

Away from ... dusty environments.
... exposure to direct sunlight.
... direct air drafts (fans, room air ducts).
... poorly ventilated areas.

Installing the Catch Trough

Ice and frost are created during normal use of the Liquid Nitrogen Cooling System (LNCS). The catch trough is designed to prevent water from dripping onto the floor creating a potential hazard when the ice and frost melt.

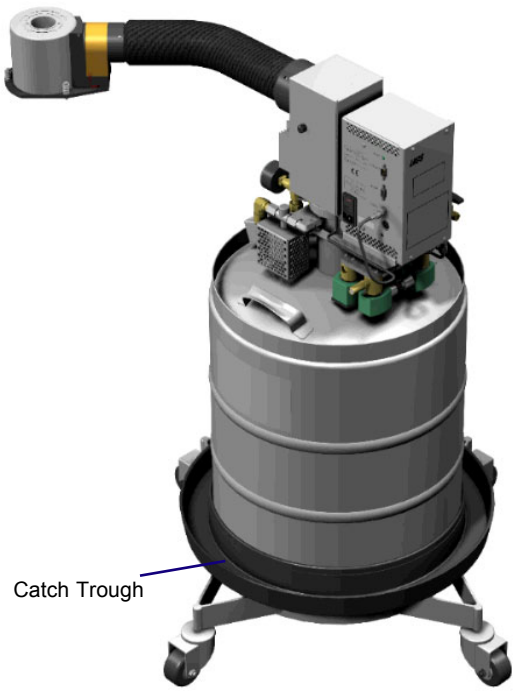
The catch trough is installed as follows:

1. Using a 5/8-inch wrench on the brass fitting, screw the plastic valve into the fitting until it is hand tight with the handle facing out.

2. Slip the catch trough down over the tank. Move the catch trough down as low as possible on the tank as shown in the figure to the right.

3. Place the stainless steel band clamp over the inside edge of the trough using the molded lip as an alignment guide. Tighten the clamp with a screwdriver to seal the trough to the tank. Do not over tighten the clamp.

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



CAUTION: During manual filling operations, do not over fill the LNCS tank causing liquid nitrogen to spill into the catch trough. Excessive amounts of LN_2 will cause the catch trough to become brittle and shatter.

Installing the LNCS

Installation of the LNCS with either the DSC Q100 or Q1000 is exactly the same. This section provides a set of instructions that you can use to install the LNCS on either DSC instrument.

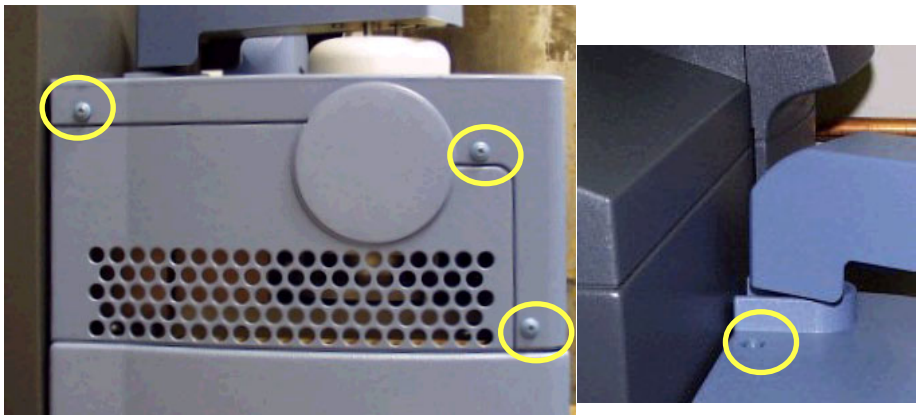
CAUTION: If your liquid nitrogen source has more than 170 kPa gauge (25 psig), then a pressure regulator must be added to ensure that no more than 25 psig is delivered to the LNCS transfer line. Failure to limit the pressure may result in damage to the fill solenoid valve, cause excessive fill times,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

Installing the Cooling Head

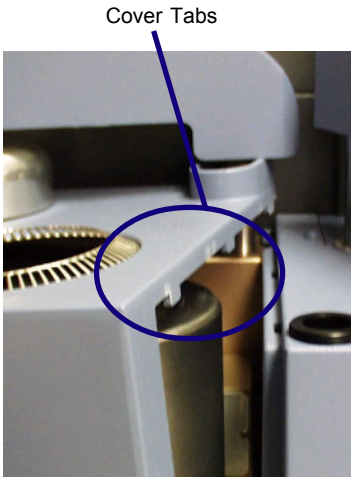
Follow the instructions below to install the cooling head on the DSC:

1. Remove the DSC lid(s). Select the **Control/Lid/Open** function on the instrument control software to raise the AutoLid from the Q100 or Q1000 cell and cause it to move out of the way.

2. Pull the plug on the side of the unit cover out to remove it. Then remove the screws attaching the cell cover to the unit cover (see the figure to the right). Three screws are located on the side (Q1000) and one is located on the top. Retain the screws.



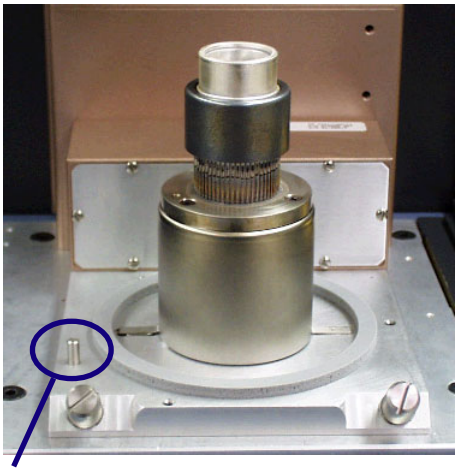
3. If your instrument has an Autosampler installed, lift up the cover to release the tabs and pull the cover towards you to remove it fully (shown in the figure below left).



If you do not have an Autosampler, you will have to remove additional screws to release the cover. Then pull the cover towards you to remove it fully. The cell will be exposed as shown in the figure on the next page.

4. If the heat exchanger hose is already attached to the LNCS dewar, you will need to loosen the supply and exhaust fittings before mounting the cooling head on the DSC cell. Refer to "Connecting the LNCS Lines" for guidance, if needed. This allows the hose to rotate freely when positioning the cooling head in the steps to follow.

5. Align the pin on the cell base (shown here) with the corresponding slot in the LNCS Cooling Head base. Carefully lower the head over the cell and make sure it is fully seated.



6. Obtain a long 3/32-inch hexagonal (Allen) wrench from the accessory kit.

7. Insert the tip of the wrench into any one of the three captive screws in the LNCS plate while holding onto the cooling head. (See the figure below.) DO NOT fully tighten the screws yet.

Installing the 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 until you feel the shoulders touch the bottom. Do not over tighten.

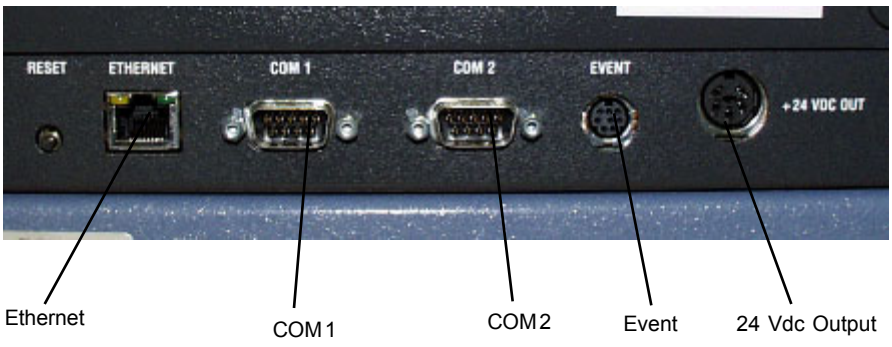
9. Slide the cover back over the cell and replace the screws removed originally.

10. Obtain access to the back of the LNCS and the back of the instrument.

11. Locate the interconnect cable. Plug one end of the cable into the 15-pin D instrument connector on the LNCS. Plug the opposite end of the cable into the port labeled COM2 on the back of the DSC instrument (see the figure below).

12. Make sure that the large hose is not sharply bent or folded. It should curve gently between the instrument and the LNCS.

13. If you loosened the supply and exhaust fittings in step 4 to allow the hose to rotate freely, retighten those fittings now.



Five Ports on Left Rear of DSC

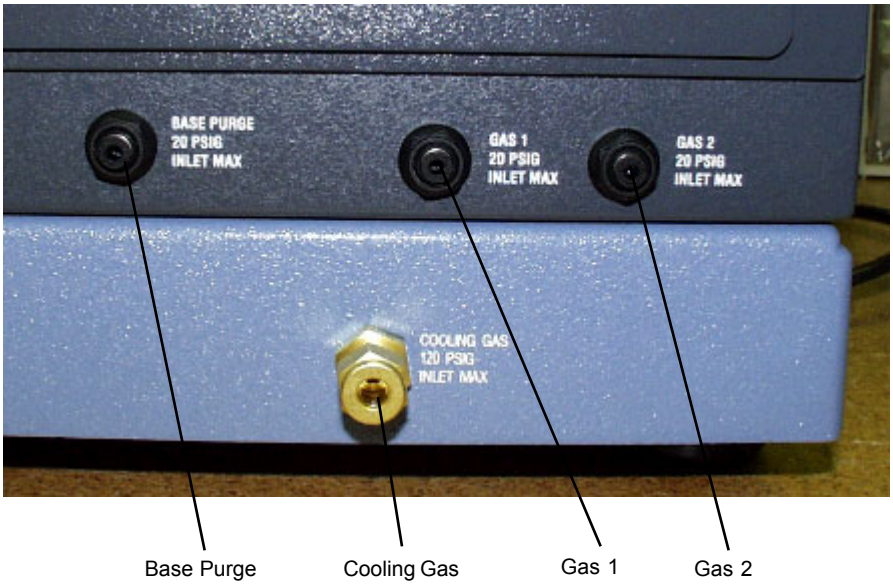
14. Check the AutoLid alignment and adjust, if needed. See the *DSC Q Series Getting Started Guide*, Chapter 3 "Aligning the AutoLid" for the procedure.

15. Follow the instructions on the next page to connect the cell base purge and the cooling gas (LNCS purge) line.

Connecting the Base and LNCS Purge Lines

Two other purges are required in addition to the standard DSC cell purge when the Liquid Nitrogen Cooling System (LNCS) is used. One purge, the Base Purge, is used to continuously purge the base of the cell. The other purge, the LNCS Purge, is used to automatically purge the interior of the LNCS cooling head when the cell is open during loading/unloading samples under Autosampler control (which is standard on the Q1000 and optional on the Q100) and during cell conditioning. Follow the instructions below to connect the lines for those purges.

1. Locate the Base Purge port. It is one of the four ports on the right rear of the instrument as shown in the figure below.



Four Ports on the Right Rear of the DSC

2. Make sure that the pressure of your gas source is regulated to 140 kPa gauge (20 psig). Dry nitrogen is the recommended gas.

3. Use 1/8-inch O.D. tubing to connect the gas source to the Base Purge. Teflon® TFE tubing is recommended. An orifice in the instrument will automatically regulate the flow rate (300 to 350 mL/min) for proper operation.

4. Locate the Cooling Gas port on the right rear of the instruments (as shown in the figure above). The LNCS Purge will be connected to that port.

5. Make sure that the pressure of your gas source for the LNCS Purge is also regulated to 140 kPa gauge (20 psig). Dry nitrogen should be used.

   NOTE: Since both the Base Purge and LNCS Purge will be exposed to temperatures below ambient, the gases used should be moisture-free. Nitrogen gas of 99.999% purity is recommended.

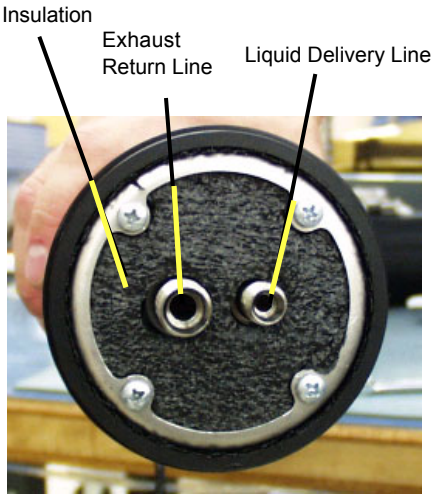
6. Use 1/4-inch O.D. tubing to connect the gas source to the Cooling Gas port on the back of the DSC instrument for the LNCS Purge. Teflon® TFE tubing is recommended. A solenoid valve, automatically regulated by the Advantage Q Series™ software, determines when the LNCS Purge is on. An orifice in the instrument automatically regulates the flow rate to (300 to 350 mL/min) for proper operation.

Connecting the LNCS Lines

After you have mounted the cooling head (also called the heat exchanger) on the DSC cell, follow these instructions to connect the supply and exhaust lines:

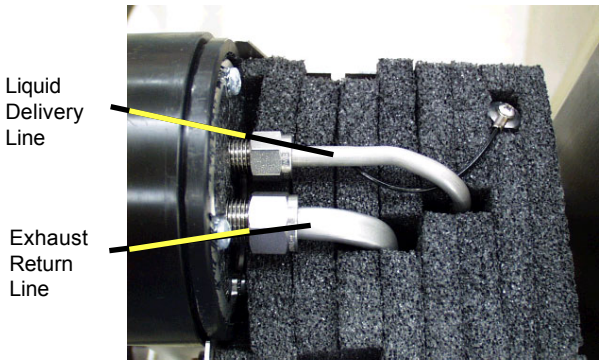
1. Loosen the captive screws on either side of the top cover of the liquid delivery tower. Pull the cover straight up to remove it.

2. Obtain the 1.8-m (6-foot) long feed hose with its attached cooling head. At the opposite end from the cooling head are two lines that need to be connected to the LNCS liquid delivery tower. The figure here identifies the two lines that will be attached.



3. Using a 9/16-inch wrench attach the smaller liquid supply line to the smaller fitting as shown in the figure below.

4. Using an 11/16-inch wrench attach the larger exhaust return line to the remaining fitting as shown in the figure below.



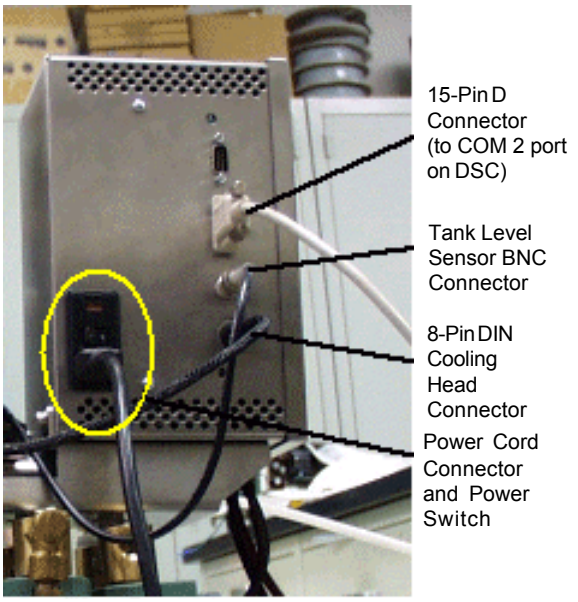
5. Replace the top cover over the liquid delivery tower.

6. Screw in the captive screws on the sides of the cover until they bottom out (finger tight).

7. Connect the 8-pin DIN cooling head connector to the port on the back of the LNCS electronics control box. See the figure to the right for the location of the connectors.

8. Connect the tank level BNC connector from the liquid delivery tower to the electronic control box, if it is not already in place.

9. Connect the 15-pin D connector cable to the instrument port on the LNCS electronic control box. The opposite end will go to the COM 2 port on the back of the DSC.



LNCS Electronic Connections

NOTE: A <HAR>-marked (harmonized) power cable meeting the standards of the country of installation is required for the European Economic Area.

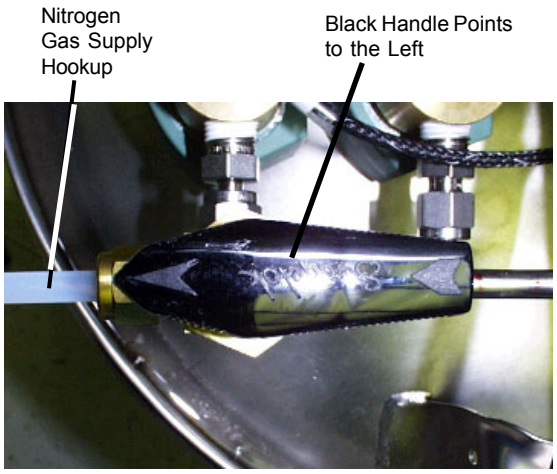
10. Plug the power cord into the electronic control box and into the power outlet.

11. Toggle the power switch on the back of the LNCS electronics control box to ON.

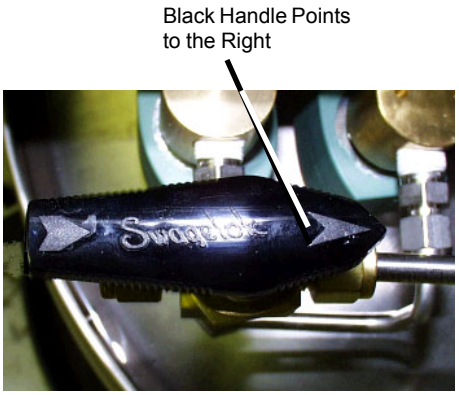
12. Connect the house nitrogen gas to the nitrogen gas supply hookup, as shown in the figure to the right, if the LNCS is to be pressurized externally.

13. Turn the black handle to the left (towards the nitrogen gas supply hookup) for *external dewar pressurization.* (See the figure to the right.) The supply line, a 50 to 70 kPa gauge (8 to 10 psig) nitrogen source, is used to control the operating pressure of the dewar to force LN2 into the heat exchanger.

WARNING: Do not use compressed air to pressurize the LNCS. Large amounts of liquid oxygen could accumulate in the dewar, creating a safety hazard.



Setup for External Pressurization



Setup for Internal Pressurization

If you want to use *internal dewar pressurization*, turn the black knob to the right, as shown in the figure to the right. No supply line needs to be connected.

You are now ready to fill the Liquid Nitrogen Cooling System. Follow the instructions in the next chapter.

Chapter 3

Filling, Conditioning, Use, & Maintenance

Overview

The Liquid Nitrogen Cooling System (LNCS) must be filled from a bulk storage tank of liquid nitrogen. There are basically two methods that can be used:

- *Autofilling* refers to the automatic filling of the LNCS from any source, whether it is filled locally from a bulk storage tank located close to the unit or filled at some remote location away from the lab. Local autofilling is the most convenient method of filling the tank. Autofill uses a function controlled from the DSC instrument control software. You can also perform an autofill sequence at a remote location, if power is available to run the unit.

- *Manual filling* is the method that must be used if no power is available to allow autofilling. Manual filling is normally done at some remote location where electricity is not accessible.

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

Connecting & Autofilling the LNCS

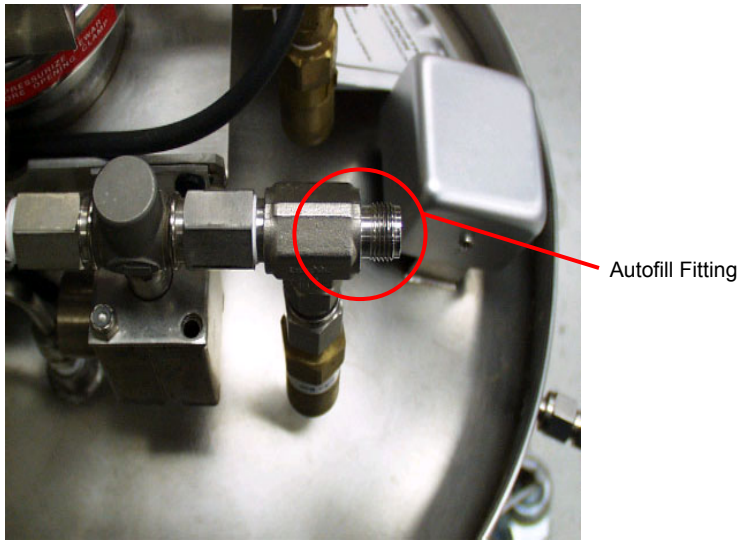
To use the local filling capability you will first need to connect the cooling accessory to a bulk source of liquid nitrogen as directed below, then fill the dewar. After the initial filling, you can set up the software to have the LNCS autofilled after experiments.

1. Arrange the low pressure bulk storage source physically close enough, within 1.8 m (6 ft), to the LNCS so that the autofill transfer tube can be easily connected between the source and the LNCS. Likewise the LNCS and the instrument need to be in close proximity to allow connection of the 1.8-m (6-ft) transfer hose.

2. Connect the LNCS for automatic filling as follows:

   a. Attach the transfer tube to the LNCS Autofill fitting (shown in the figure to the right).

   b. Attach the other end of the transfer tube to the bulk storage container using the union and adapter fitting (provided in the accessory kit).



3. Install the cooling head on the DSC as directed in Chapter 2, if it is not already installed. The cooling head must be in place on the cell and the cell must be operational before filling the LNCS.

4. Turn on the power to the LNCS and the instrument.

5. Fill the dewar with liquid nitrogen before beginning an experiment following the directions in the next section for "Initial Autofilling."

Initial Autofilling

The LNCS must be filled before cooling experiments can be performed on DSC Q Series™ instruments. Follow the instructions in this section to fill the LNCS.

Using the Thermal Advantage Q Series Explorer, connect to the desired instrument. Select **Control/LNCS/Fill** from the instrument control main menu. The LNCS will be filled automatically.

The autofill will shut off when the dewar is full, the bulk storage tank is empty, or the LNCS tank pressure is below 1 psig for more than 1 minute. Once the dewar has been filled initially, you can set up programmed autofilling as directed in the next section.

Programmed Autofilling

"Autofilling" also refers to the automatic refilling of the LNCS from the bulk storage tank between runs. This section tells you how to set up the LNCS and the connected instrument to allow autofilling.

The Q Series instruments automatically control the LNCS, which regulates pressure, supplying liquid nitrogen to the cooling head.

To automatically refill the LNCS with liquid nitrogen after an experiment on a Q Series instrument is completed, access the **Tools/Instrument Preferences/DSC Page**, select **LNCS Autofill if below**, then enter the desired percent.

> NOTE: The LNCS is normally filled from a bulk tank located near the unit. If you need to fill your LNCS dewar with liquid nitrogen from a remote source (*i.e.*, source not located near your unit), follow the directions beginning on the next page.

Remote Filling of the LNCS

There is also the ability to perform an autofill sequence at a remote location (away from the controller and instrument), if power is available to run the unit. Depressing the Fill button on the side of the electronics control enclosure for 3 seconds or longer initiates an autofill. This same button will reset the LNCS if depressed for less than three seconds.

If power is not available at the remote filling location, the LNCS can be filled manually.

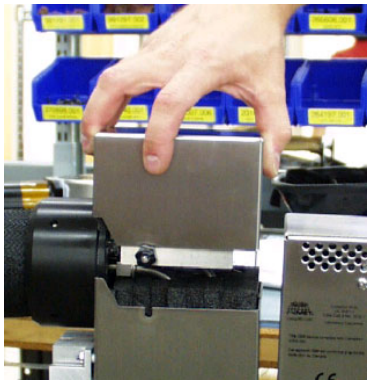
This section describes both the automatic and manual remote filling methods.

Remote Autofill

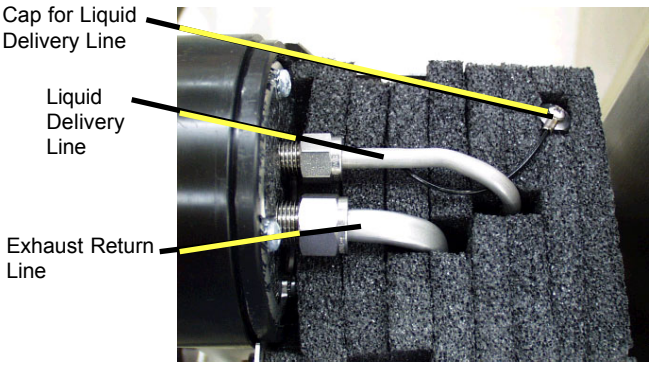
Follow the directions in this section to fill the LNCS automatically at a remote location:

1. Turn off the power to the LNCS.

2. Loosen the captive screws on either side of the top cover for the liquid delivery tower. Pull the cover straight up to remove it as shown in the figure to the right.



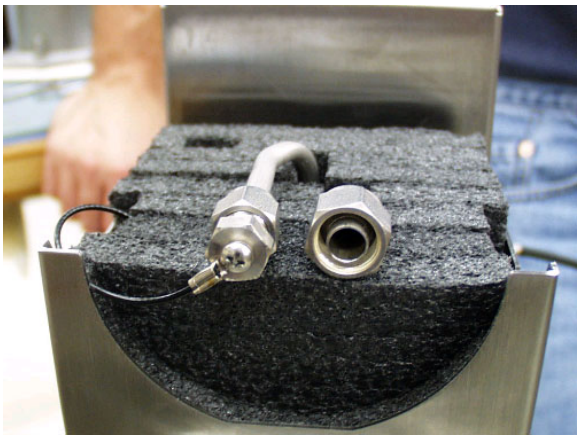
3. Using a 9/16-inch wrench remove the smaller liquid delivery line from its fitting as shown in the figure below.



4. Using an 11/16-inch wrench remove the larger exhaust return line from the remaining fitting as shown in the figure immediately above.

5. Pull the feed hose off the liquid delivery tower. Located in the top of the tower is the cap for the liquid delivery line (shown in the figure above left). Remove the cap with its attached wire from the tower insulation.

6. Screw the cap onto the liquid delivery line. See the figure to the right.



WARNING: If the feed hose is disconnected from the LNCS, the fitting on the liquid supply line must be capped (see steps 5 and 6) to prevent liquid nitrogen from spraying out of the fitting during the fill process.

7. Disconnect the house nitrogen gas supply tube, if connected.

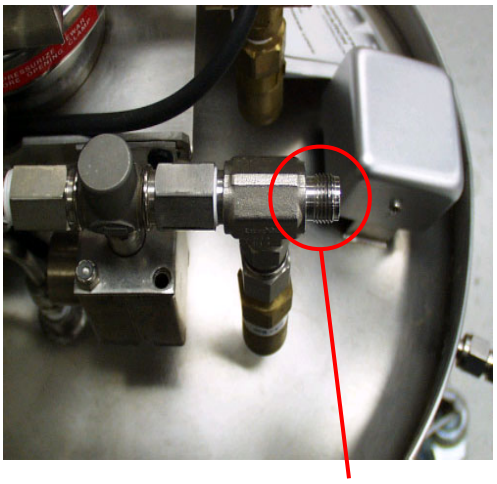
8. Disconnect the 15-pin D connector cable at the LNCS. Disconnect the 8-pin DIN cooling head connector from the electronic control box.

9. Unplug the power cord, but leave it attached to the LNCS.

10. Roll the LNCS to the location of the bulk storage source and plug the power cord into the closest power outlet. Turn the power switch ON.

11. Make sure that the bulk storage source that will be used for filling the LNCS is a low pressure (maximum 25 psi) container.

12. Connect the transfer hose from the bulk source to the autofill fitting shown in the figure here.



Autofill Fitting

13. Open the valve on the bulk storage source.

Fill Button

14. Depress and hold the Fill button (shown in the figure to the left) on the LNCS control box for 3 seconds to initiate the autofill. The filling will stop automatically when the dewar is full.

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

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

15. After the autofill has completed, allow sufficient time for any liquid remaining in the transfer tube to vaporize.

16. Close the valve on the nitrogen bulk storage tank and immediately disconnect the transfer tube from the bulk source.

17. Disconnect the transfer hose from the LNCS autofill valve, turn off the power switch, and unplug the power cord.

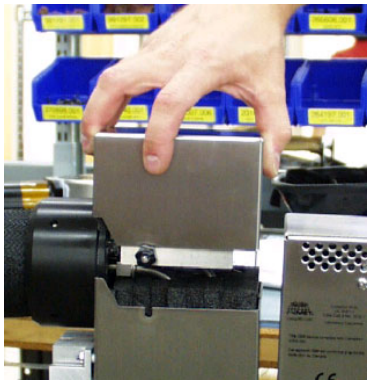
18. Return the LNCS to its location near the analysis instrument, and reconnect the cooling accessory by reversing steps 1 to 9.

Remote Manual Fill

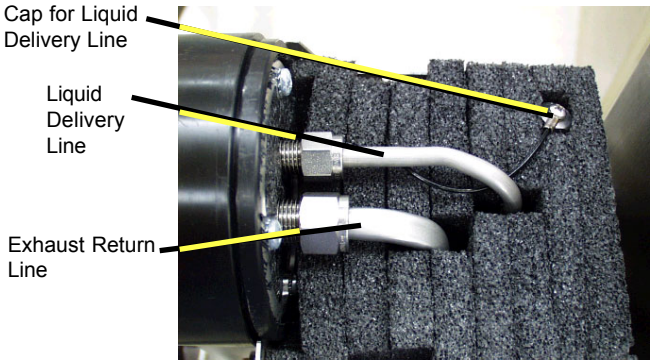
Follow the directions in this section to fill the LNCS manually at a remote location:

1. Turn off the power to the LNCS.

2. Loosen the captive screws on either side of the top cover for the liquid delivery tower. Pull the cover straight up to remove it as shown in the figure to the right.



3. Using a 9/16-inch wrench remove the smaller liquid delivery line from its fitting as shown in the figure below.

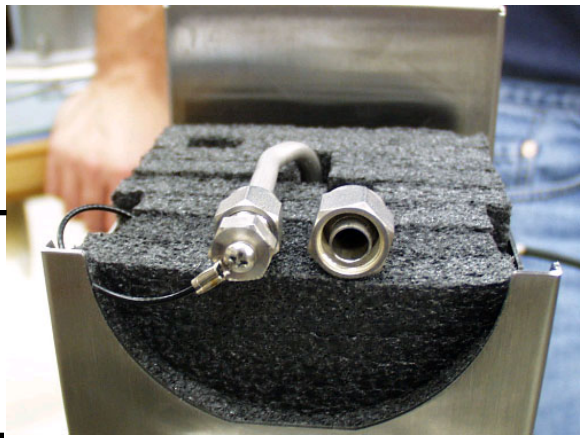


4. Using an 11/16-inch wrench remove the larger exhaust return line from the remaining fitting as shown in the figure immediately above.

5. Pull the feed hose off the liquid delivery tower.

6. Locate the cap for the liquid delivery line (shown in the figure above left) in the top of the tower. Remove the cap with its attached wire from the tower insulation.

7. Screw the cap onto the liquid delivery line. See the figure to the right.



WARNING: If the feed hose is disconnected from the LNCS, the fitting on the liquid supply line must be capped (see steps 5, 6 and 7) to prevent liquid nitrogen from spraying out of the fitting during the fill process.

8. Disconnect the house nitrogen gas supply tube, if connected.

9. Disconnect the 15-pin D connector cable at the LNCS. Disconnect the 8-pin DIN cooling head connector from the electronic control box.

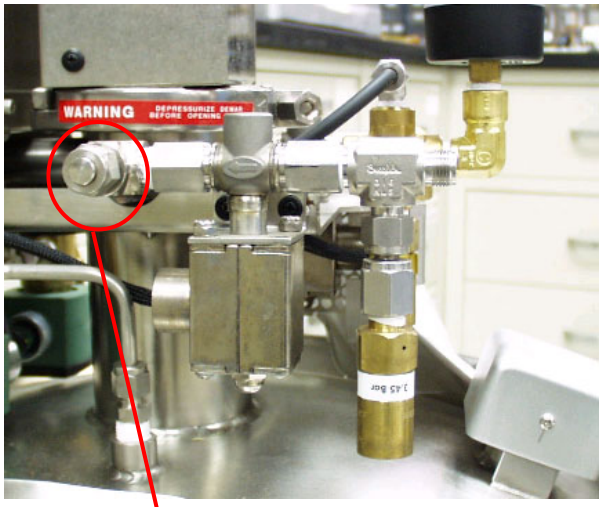
10. Unplug and disconnect the power cord from the LNCS.

11. Roll the LNCS to the location of the bulk storage source.

12. Make sure that the bulk storage source that will be used for filling the LNCS is a low pressure (maximum 25 psi) container.

13. Using a 11/16-inch wrench, remove the cap from the manual fill fitting (shown in the figure to the right). Connect the transfer hose from the bulk source to the manual fill fitting.

14. Place the dewar on a scale, if one is available, so that you can monitor the weight to determine when the dewar is full.



Manual Fill
Capped Fitting

15. Open the valve on the bulk storage source and leave it open until the dewar reaches a filled weight of 87 kg (193 lbs).

WARNING: If liquid begins to spill from the vent during filling, stop the filling process immediately by closing the valve on the bulk source. This must be done quickly to prevent freeze damage to the unit.

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

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

16. Close the valve on the nitrogen bulk storage tank.

17. Allow sufficient time for any liquid remaining in the transfer tube to vaporize.

18. Disconnect transfer hose from the manual fill valve and replace the cap using the wrench to tighten snugly (do not over tighten).

19. Return the LNCS to its location near the analysis instrument, and reconnect the cooling accessory by reversing steps 1 to 10.

Starting the LNCS

Once the LNCS has been properly installed, follow the steps below to set up the instrument parameters and condition the LNCS-DSC system for optimum performance.

1. Verify the correct cooler type (*e.g.*, LNCS) on the **Tools/Instrument Preferences/DSC** Page of the DSC instrument control software.

2. Verify that a source of dry nitrogen is connected to the base purge and cooling gas (LNCS) purge. Select the gas to be used with the Gas 1 port on the back of the DSC instrument (see NOTE below).

   NOTE: Dry nitrogen is used for the base purge and LNCS purge. But, if the starting temperature is below ambient, helium should be used for Gas 1 (cell purge). If the starting temperature is above ambient, nitrogen may be used.

3. Dry the LNCS system before turning on the LNCS by following Step 1 of the conditioning procedure found in the next section, "Conditioning the LNCS."

4. Verify that the post-test conditions (accessed through the **Procedure Page** by clicking the **Post Test** button) are set as desired. A temperature window above ambient should be used to prevent the cell from cooling down between experiments (*e.g.*, typical values are 35 to 50°C). Once these conditions are verified, select **Go to Standby Temp** from the **Control** menu to invoke the standby temperature set on the **Tools/Instrument Preferences/DSC Page**.

   NOTE: The DSC cell should be covered when not loading samples and should not be opened below ambient temperatures.

5. Proceed to Step 2 of the conditioning procedure found in the next section, "Conditioning the LNCS" to further stabilize the DSC-LNCS system after installation. This cyclic experiment allows the DSC-LNCS system to stabilize resulting in optimized baseline and calibration.

6. Recalibrate the DSC after conditioning the system.

   NOTE: When setting up experiments, be sure to verify the post-test conditions. A temperature window above ambient should be used to prevent the cell from cooling below ambient between experiments.

Conditioning the LNCS System

Each time the LNCS heat exchanger is installed on the DSC the following conditioning procedure should be run before calibration and experiments are performed. The first step of conditioning is used when the system is first installed and periodically thereafter to dry the system to remove moisture in the DSC cell and heat exchanger BEFORE turning on the LNCS. The second step is used to stabilize the DSC–LNCS system by cycling the system to optimize baseline performance.

Step 1: Drying the System

Follow the instructions below:

1. Verify that the DSC cell is empty and cover the cell. If an AutoLid mechanism is present, verify that the lids are seated properly. (Refer to "Aligning the AutoLid in the DSC Q Series Getting Started Guide or in the online help for instructions to align the lid, if needed.)

2. Access the **Tools/Instrument Preferences/DSC Page** of the DSC instrument control software. Verify that the correct cooler type (LNCS) is selected, check "Leave LNCS on," and verify the desired "Standby Temperature."

3. Using the DSC instrument control software, access the **Experimental View Summary Page**. Select the "Standard" mode, then select the "Cell/Cooler Conditioning" test template from the list. This test is performed with the LNCS off.

4. Click on the **Procedure Page**.

5. Verify the default conditions of 120 minutes at 75°C and select **Apply**. These conditions are suitable for typical situations.

6. Access the **Post Test Parameters** window and enter a temperature range window of 35 to 50°C to return the cell to slightly above ambient. Once the LNCS is operating, it is very important that the cell is always kept at or slightly above ambient temperature before and after experiments.

7. Start the experiment.

8. Upon completion of this experiment, the base and cell purges must remain on continuously. If the purges do not remain on, the atmospheric moisture will contaminate the system and, depending on the time involved and relative humidity, the procedure may have to be repeated.

Step 2: Stabilizing the System

The following cyclic experiment is performed after the first step in order to allow the DSC-LNCS system to stabilize, resulting in optimized baselines and calibration.

1. Select **Control/LNCS/Cool** from the menu. This will enable the LNCS and begin cooling the cell. Once the LNCS has started, the flange temperature will cool rapidly to its operating temperature.

2. Verify the instrument preferences and post-test conditions as outlined in steps 2 and 6 on the previous page.

3. Verify that the cell is emptied and cover the cell.

4. Observe the **Signal Display** pane. Verify that "Set Point Temperature" displayed is at the midpoint value of the Temperature Range specified on the **Post Test Parameters** window. This indicates that the post test temperature control is active. If the post test temperature control is not active (*i.e.*, the "Set Point Temperature" reads 0.00°C), select **Go to Standby Temp** from the **Control** menu to invoke the standby temperature set on the **Tools/Instrument Preferences/DSC Page**.

5. Create and save the following "Custom" method:

    1 Data Storage On
    2 Equilibrate 50°C
    3 Isotherm 60 minutes
    4 Mark end of cycle
    5 Equilibrate 300°C
    6 Mark end of cycle
    7 Isotherm 30 minutes
    8 Mark end of cycle
    9 Equilibrate –180°C
    10 Mark end of cycle
    11 Isotherm 10 minutes
    12 Mark end of cycle
    13 Ramp 20°C/min to 300°C *(continued on next page)*
    14 Mark end of cycle
    15 Isotherm 10 minutes
    16 Repeat segment 8 for 7 times

6. Start the experiment created in step 5. The flange temperature must be below 100°C when operating an LNCS. If the run is started when the flange is above 100°C, then an error message will be posted and the run will be terminated. During normal operation the flange temperature should be less than –145°C at the start of a run.

After conditioning the LNCS (by performing both the drying and stabilization steps), evaluate the last baseline run in the method above for any artifacts. Calibrate the DSC *before* running experiments using the LNCS. See the DSC online help for details.

Using the LNCS

It is best to start the LNCS (see page 40) before you run an experiment. This allows the LNCS to stabilize and will prevent samples from being exposed to cryogenic temperatures prior to starting a run.

Starting an Experiment

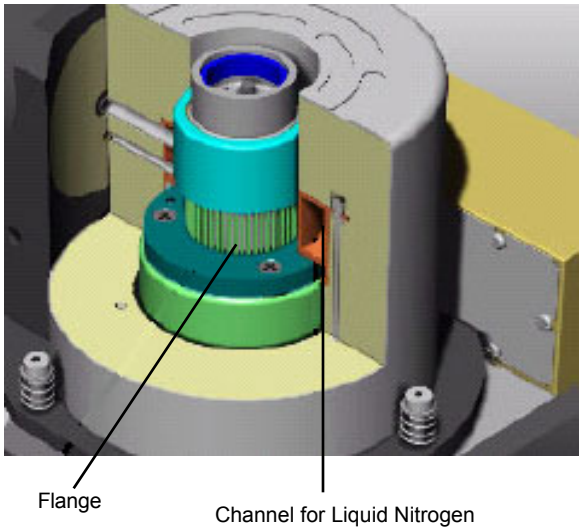
Before you start the experiment, ensure that the DSC is connected with the controller, the standard and base purge gases are connected, and that you have entered all necessary information through the instrument control software.

> NOTE: Once the experiment is started, operations are best performed at the computer keyboard. The DSC is very sensitive to motion and might pick up the vibration caused by touching a key on the instrument touch screen.

Start the experiment by selecting **Start** on the instrument control software or by touching the START key on the instrument touch screen (Q100 or Q1000). When you start the instrument, the system automatically runs the experiment to completion.

LNCS Starting Conditions

If you are using the Liquid Nitrogen Cooling System (LNCS), the run will start when the flange (shown here), which is part of the cell structure, has reached a temperature below –160 °C and when the system has detected adequate liquid nitrogen is present. The cell temperature is then set to 20°C and the experimental method is started.



Guidelines When Using the LNCS

Once the LNCS is properly installed, the system conditioned and calibrated, the following guidelines should be maintained during standard experimental operation.

- A dry, moisture-free gas source is required for the cooling gas (LNCS) purge and the base purge when using the LNCS, in addition to the standard cell purge gas. Dry nitrogen is recommended for this purge. These gases must remain on continuously. If they do not, the atmospheric moisture will enter and contaminate the system.

- The LNCS Purge is automatically on whenever the cell is opened by the AutoLid to prevent moisture from entering the system. (NOTE: This does not function when the cell lid is manually opened as it is on the DSC Q10.) It is strongly recommended that the cell lids be in place anytime that a sample is not being actively loaded or unloaded. Turn the LNCS purge on using **Control/Air Cool/On** prior to removing the lids. Turn the LNCS purge off after the lids are in place.

- *Important:* If you are planning to run subambient experiments, use helium as the purge gas. If you are using the LNCS for rapid cooling above ambient (*i.e.*, isothermal crystallization), then nitrogen may be used as a purge gas.

NOTE: Please make sure that you run your experiments with the same gas that you used to calibrate the system. For example, if you calibrate using nitrogen, make your runs with nitrogen.

- Access the **Tools/Instrument Preferences/DSC Page** of the DSC instrument control software. Verify that the correct cooler type (LNCS) is selected and check "Leave LNCS on." Check the "LNCS Autofill if below" option, then enter a percentage to automatically fill the LNCS, if desired. This indicates that you want the LNCS to automatically fill at the end of an experiment when the level of liquid nitrogen falls below the specified percent. The fill process, when activated, will fill to completion before advancing to the next scheduled run. If left unchecked, you will need to manually fill the LNCS when needed. (Default = checked, 40%)

- When setting up experiments, be sure to verify the post-test conditions. The temperature window should be enabled and a temperature range above ambient should be used to prevent the cell from cooling down between experiments.

- When setting up an Autosampler sequence, access the **Instrument Preferences/Autosampler Page** and select the desired sequence-end option for the LNCS.

- *Important*: DO NOT open the DSC cell at below ambient temperatures to prevent frost and moisture buildup in the cell. If this occurs, the conditioning and calibration steps may have to be repeated.

- *Important*: Once the DSC-LNCS system has been conditioned, it is recommended that you do NOT turn off the LNCS between runs, if the best possible baseline performance is desired.

- *Important:* Operating without an effective base purge, allowing the cell to remain at the lower temperature limit without heater power (*e.g.*, without post-test conditions) for extended periods of time, and/or removing the LNCS from the cell when the flange temperature is below ambient can result in excessive moisture in the cell and requires extended time for drying such as performing Step 1 of the conditioning procedure found in the section, "Conditioning the LNCS."

WARNING: Do not exceed 100°C with the LNCS cooling head installed and the LNCS power off. Serious damage to the cooling head could occur.

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

NOTE: Once the cooling flange reaches operating temperature, it condenses any moisture present. If the initial moisture level is too high, or if the atmosphere moisture subsequently entering the heat exchanger enclosure is not minimized, then artifacts can be observed in the heat flow signals. Typically, but not exclusively, the artifacts are observed between 0 and 100°C, which increase in intensity over time.

Maintaining the LNCS

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the online documentation installed with the instrument control software for further information.

WARNING: Because of the high voltages in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

CAUTION: Before using any cleaning or decontamination method except those recommended by the manufacturer, users should check with the manufacturer that the proposed method will not damage the equipment.

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

- Cleaning
- Fuse replacement
- Graphite gasket replacement.

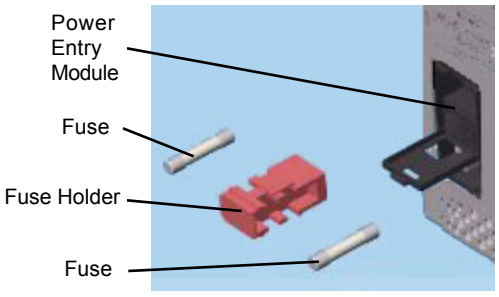
Cleaning the LNCS

You can clean the LNCS 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.

Replacing the Fuses

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



1. Turn the cooling accessory off and remove the power cord.
2. Insert a small screwdriver at the edge of the power entry module door and pry it open.
3. Insert the screwdriver on the edge of the fuse holder to pull it out of the instrument.
4. Remove old fuses and replace the fuses only with the type and rating indicated on the instrument's rear panel.

5. Place fuse holder back into opening and push the door shut.

6. Replace the power cord and turn the unit back on.

Replacing the Graphite Gasket

Inside the LNCS cooling head are several items that function to provide a tight seal between the DSC cell and the cooling accessory. If you find that the cooling performance of your unit begins to produce less than desired results, you may need to check the graphite gasket inside the cooling head and replace it, if needed, by following these instructions:

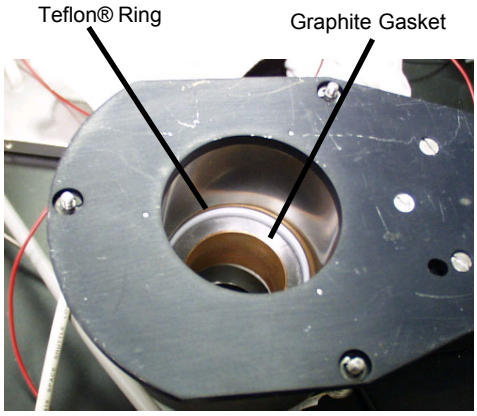
1. Turn off the power to the unit and wait until the flange temperature is above ambient.

2. Remove the cooling head. See the installation instructions beginning on page 17, if needed.

2. Turn the cooling head upside down as shown in the figure here.

3. Inspect the graphite gasket for any tears, holes, or signs of wear. Also inspect the cooling flange on the DSC cell for any graphite residue. Replace the gasket, if needed, by following the next several steps.

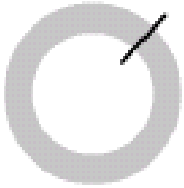
4. Using a small flat head screwdriver, pry out the white Teflon® ring that holds the graphite gasket in place. The ring is slotted to allow enough flexibility for removal.



Inside the Cooling Head

5. Remove the damaged graphite gasket and discard it.

6. Obtain the new gasket and a pair of scissors. Carefully make a cut across the diameter of the gasket on one side to allow installation. (It is shipped uncut to avoid damage during shipping.)


Snip with scissors to slot gasket

7. Carefully press the new gasket down into the cooling head, taking care not to damage the thin material. Allow the edges of the gasket to slide into the groove located inside the cooling head for that purpose.

8. Replace the white Teflon® ring, with the beveled side facing out, so that it snaps back into place and secures the gasket.

9. Install the cooling head on the instrument again and turn the power on.

10. Check the AutoLid alignment and adjust, if needed. See the *DSC Q Series Getting Started Guide*, Chapter 3 "Aligning the AutoLid" for the procedure.

Replacement Parts

Replacement parts for the LNCS that are available from TA Instruments. See the table below when ordering parts.

Part Number	Description
970408.901	Cooling Head Assembly
271282.001	Power Supply
970250.901	Printed Circuit Board, Control, LNCS
271562.001	Fuse, 2.5A, 250V
970322.901	Autofill Valve
970323.901	Autofill Vent Valve
970324.901	Pressure Build Valve Assembly (L11)
970325.901	Pressure Build Vent Valve Assembly (L12)
970326.901	Pressure Control Valve Assembly (L13)
970327.901	Pressure Control Vent Valve Assembly (l14)
200121.002	345 kPa gauge(50 psig) Pressure Relief Valve, Fill Tube Protection
200121.001	90 kPa gauge (13 psig) Pressure Relief Valve, Dewar Protection
970374.001	Gasket, Graphite, Heat Exchanger
970076.001	Centering Ring Heat Exchanger
970418.901	Dewar Cap Assembly

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