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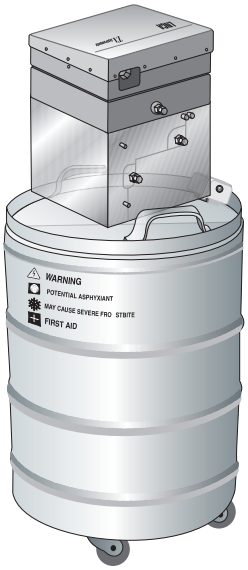

Liquid Nitrogen Cooling Accessory CE

Operator's Manual

PN 891300.001 Rev. D (Text and Binder)
PN 891300.002 Rev. D (Text Only)
Issued November 1999

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.

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.**

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

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Safety

This equipment has been designed to comply with the following standards on safety:

- IEC 1010-1/1990 + A1/1992 + A2/1995
- IEC 1010-2-010/1992 + A1/1996
- EN 61010-1/1993 + A2/1995, Installation Category II.
- EN 61010-2-010/1994
- UL 31031-1, First Edition.

CE Compliance

In order to comply with the Electromagnetic Compatibility standards of the European Council Directive 89/336/EEC (EMC Directive) and Directive 73/23/EEC on safety as amended by 93/68/EEC, the following specifications apply to the LNCA CE:

- *Safety:*
  EN 61010-1/1993 + A2/1995 Installation Category II
  EN 61010-2-010/1994

- *Emissions:*
  EN 55022: 1995, Class B (30–1000 MHz) Radiated
  EN 55022: 1995, Class B (0.15–30 MHz) Conducted

- *Immunity:*
  EN 50082-1: 1992, Electromagnetic Compatibility—Generic immunity standard Part 1. Residential, commercial, and light industry.
  - — IEC 801-2: 1991. Electrostatic discharge.
  - — IEC 801-3: 1984. RF field.
  - — IEC 801-4: 1988. Electrical fast transients/burst.

Safety

(continued)

Instrument Symbols

The following label is displayed on the Liquid Nitrogen Cooling Accessory for your protection:

Symbol	Explanation
	This symbol, located on the clamp on the neck of the LNCA CE tank, indicates the presence of a hazardous voltage indise the LNCE CE. Do not remove the clamp securing the cap to the dewar.

Please heed this label and take the necessary precautions when dealing with this part of the accessory. The *Liquid Nitrogen Cooling Accessory Operator's Manual* contains cautions and warnings that must be followed for your own safety. Refer to the information in this section for specific information related to safety.

Safety

(continued)

Safe Handling of Cryogenic Materials

Liquid nitrogen is used as a cooling agent in many thermal analysis tests. Because of its extremely low temperature (–196°C) it will burn skin. You **must** use extreme care when working with liquid nitrogen or other cryogenic materials.

Liquid nitrogen can:

1. Cause serious skin burns.
2. Replace the air in the room you are in.
3. Generate very high pressures if trapped in lines or containers.

Handling Liquid Nitrogen

1. Wear goggles or a face shield; gloves that are easily removed; and high-topped shoes with pant legs *outside* the tops for extra protection.

2. Transfer the liquid slowly to prevent thermal shock to the container and excessive turbulence to the liquid nitrogen. If liquid nitrogen is poured into a container that is at room temperature, the liquid nitrogen will boil violently. Use extreme caution to prevent the boiling liquid from contacting you.

Safety

(continued)

3. Use only approved low temperature containers.

4. Make sure liquid nitrogen containers are vented to prevent pressure buildup.

If Burned by Liquid Nitrogen

1. Flood the area (skin or eyes) IMMEDIATELY with large quantities of cool water, then apply cold compresses.

2. See a doctor IMMEDIATELY if the skin is blistered or if the liquid nitrogen came in contact with your eyes.

Room Ventilation

Liquid nitrogen evaporates quickly at room temperature and could replace the air in a room. Only use liquid nitrogen in a well ventilated room. Important—see the Warning on page *x*.

Oxygen Absorption

Liquid nitrogen absorbs oxygen from the air. It is possible for the purity of liquid nitrogen to change as it evaporates from a container. If you suspect that a lot of liquid nitrogen has evaporated, the remaining liquid should be analyzed for oxygen content before being used for any purpose in which high oxygen content is dangerous.

Safety
(continued)

Pressure Buildup

Liquid nitrogen should not be stored in a sealed container, as tremendous pressure could result and an explosion is possible.

The LNCA CE is designed to always be vented to the room when not supplying liquid nitrogen to the instrument. The pressure buildup in the LNCA CE when it is supplying liquid nitrogen to the instrument is limited by the controller. Also, a pressure relief valve is designed into the system.

!WARNING

Never plug or cap the transfer tube on the LNCA CE. Always make sure the LNCA CE system is installed correctly.

!WARNING

Never allow liquid nitrogen to be trapped in the supply line from the instrument to the LNCA CE or the fill line from the bulk storage tank to the LNCA CE.

The sequence for opening and closing valves is important to prevent trapping of liquid nitrogen in the transfer tube. When the Autofill feature is connected, it is important that the manual valve on the bulk storage tank *never* be closed unless the bulk storage tank is empty or the LNCA CE has been turned off with the power cord removed for at least 15 minutes. This time allows the liquid nitrogen to vaporize before the area between the solenoid valve and the valve on the bulk storage tankis sealed. A pressure relief valve is connected to the transfer line as an added safety measure.

Safety

(continued)

!WARNING

Never remove the LNCA CE transfer tube without closing the bulk storage tank valve first.

When connecting and removing the LNCA CE transfer tube, remember to wear goggles and gloves.

!WARNING

Do not use high pressure bulk tanks. The LNCA CE is designed for lower pressure bulk tanks. Using high pressure tanks will damage gauges, causing the LNCA CE to work improperly, and raise the potential for injury.

Water Condensation

The LNCA CE surfaces get cold during use of the LNCA CE for both filling and supplying liquid nitrogen to the instrument. The cold surfaces cause condensation and, in some cases, frost to build up. This condensation may drip to the floor. Provisions to keep the floor dry should be made. If any moisture does drip to the floor, be sure to clean it up promptly to prevent a slipping hazard.

Safety

(continued)

Electrical Safety

High voltages (120 Vac) are present in this instrument. Only qualified service personnel should remove covers and make repairs.

WARNING

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

WARNING

Hazardous voltage is present inside the LNCA CE. Do not remove the clamp securing the cap to the dewar. There are no user serviceable parts inside the LNCA CE. Call TA Instruments for service.

!WARNING

Potential Asphyxiant

Liquid nitrogen can cause rapid suffocation without warning.

Store and use in an area with adequate ventilation.

Do not vent LNCA CE container in confined spaces.

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

The warning above applies to the use of liquid nitrogen. Oxygen depletion sensors are sometimes utilized where liquid nitrogen is in use. Please refer to the rest of this section of the for more detailed instructions regarding safety in the use of the LNCA CE.

Chapter 1: LNCA CE Overview

Introduction

The LNCA (Liquid Nitrogen Cooling Accessory) CE is a universal cooling accessory for use with TA Instruments Analyzers. It can be used with the Differential Scanning Calorimeter (DSC) models 2010 CE and 2920 CE.

The LNCA CE has been designed for automatic refilling from a low pressure 170 kPa maximum (25 psi) bulk storage tank that can be located within 6 feet of the LNCA CE. You can also fill the LNCA CE manually by disconnecting it from the instrument and moving it to a bulk storage source.

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

Specifications

The specifications in Tables 1.1 and 1.2 apply to the Liquid Nitrogen Cooling Accessory.

Table 1.1
LNCA CE Technical Specifications

LNCA CE liquid nitrogen capacity	50 Liters
Size	115 cm (45 in.) high by 48 cm (19 in.) in diameter
Power requirements	120 volts at 3.5 amps, 50–60 Hz. Use step-down transformer when using 230 Vac power source.
Weight	30 kilograms (65 pounds)
Cooling capacity	–150°C with all instruments
Pressure relief	70 kPa (10 psi) relief valve 345 kPa (50 psi) on fill line
Pressure gauge	0–210 kPa (0–30 psi)
Liquid nitrogen feed tube	180 centimeters (6 ft) insulated from LNCA CE to instrument.

(table continued)

Table 1.1 (continued)

Liquid nitrogen fill tube	180 centimeters (6ft) insulated (identical to feed tube) from LNCA to bulk storage. Supplied with union and adapter for bulk storage connection.
LNCA CE fill modes	Automatic—bulk storage within 180 cm (6 ft) of LNCA CE Manual—remote filling at bulk storage location
Bulk storage tank	Use low pressure supply tank only. Recommended filling pressure is 140–170 kPa (20–25 psi).

Table 1.2 shows the performance specifications for LNCA CE/DSC cooling. These specifications are based on operation with a full and pressurized liquid nitrogen tank.

NOTE: Only values with tolerances or limits are guaranteed data. Values without tolerances are for information only.

Table 1.2 LNCA CE/DSC Performance Specifications

Temperature Range	–150°C to 725°C with the DSC Cooling Accessory in place

Linear Cooling Rates

Rate	Temperature	Peak-to-Peak Baseline Noise
Up to 5°C/min	400°C to –150°C	<0.120 mW
Up to 20°C/min	400°C to –50°C	<0.120 mW
20°C/min and up	Depends on the temperature range of interest. May be higher accompanied by increased curvature of the baseline of the cooling curve.	

Linear Heating Rates

Rate	Temperature	Peak-to-Peak Baseline Noise
5 to 20°C/min	–100°C to 400°C	<0.120 mW

Some decrease in capacity for fast cooling rates may be observed as the level of liquid nitrogen in the LNCA CE tank nears the bottom. Under heavy demands, such as quench cooling on an unpressurized tank (first use since filling), a small delay will be observed while the tank heaters bring the LNCA CE to full operating pressure.

You can use the Pressure Gauge as an indicator of the pressure in the LNCA CE when running a test.

NOTE: The LNCA CE vents to atmosphere with a pressure reading of zero if no filling or testing is currently in progress.

Theory of Operation

Nitrogen gas at –196°C can provide some cooling; however, to provide enough cooling to reach low temperatures or to cool rapidly, liquid nitrogen is also needed. The heat of vaporization of liquid nitrogen speeds the cooling process. An ideal cooling source would provide cold gas for low cooling demands, a mixture of gas and liquid for intermediate cooling, and liquid alone for maximum cooling. The LNCA CE approaches this ideal by the use of pressure to control the amounts of gas and liquid delivered to the cell.

The LNCA CE uses up to three selectively switched 125-watt heaters to vaporize the liquid nitrogen and obtain required pressures of up to 40 kPa (6 psi). As the liquid is heated, vapor is collected and is routed through an orifice and then past a mixing jet. At the same time, liquid from the bottom of the tank is forced through a second tube to the mixing jet (Figure 1.1).

The ratio of liquid to gas at the mixing jet is a function of tank pressure, which is controlled automatically by the heater circuitry. When a small amount of cooling is needed, the pressure is low, the gas flow is slow, and no liquid is forced up through the mixing jet. When a large amount of cooling is needed, the pressure is high, the gas flow is rapid, and large amounts of liquid are forced up through the mixing jet and mixed with the gas. The liquid nitrogen mist that is created continues out the transfer tube to the instrument. At intermediate values of cooling, less liquid is mixed with the gas.

The exact control operation will be described in the chapter that pertains to the specific instrument being used.

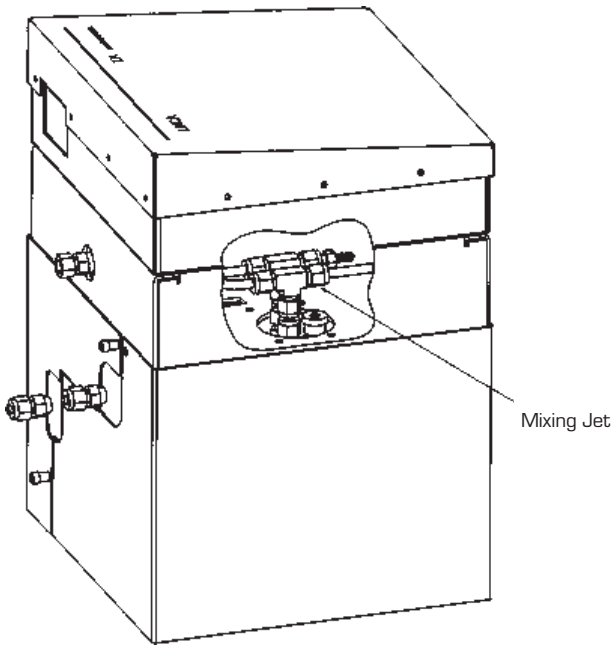

Figure 1.1
Mixing Jet

The operation of the LNCA CE is very simple. This is the sequence of operation (refer to Figure 1.2 for an illustration of the numbered parts):

1. Instrument requests coolant.
   a. Heater in LNCA CE is turned on.
   b. Feed valve #5 opens.
   c. LNCA CE fill valve #7 is closed.
   d. Vent valve #6 is closed.

2. Instrument no longer requests coolant.
   a. Heater in LNCA CE is turned off.
   b. Feed valve #5 closes.
   c. With Autofill selected, LNCA CE fill valve #7 is opened.
   d. Vent valve #6 is opened.

For more detailed information, refer to the chapter on the specific instrument being used.

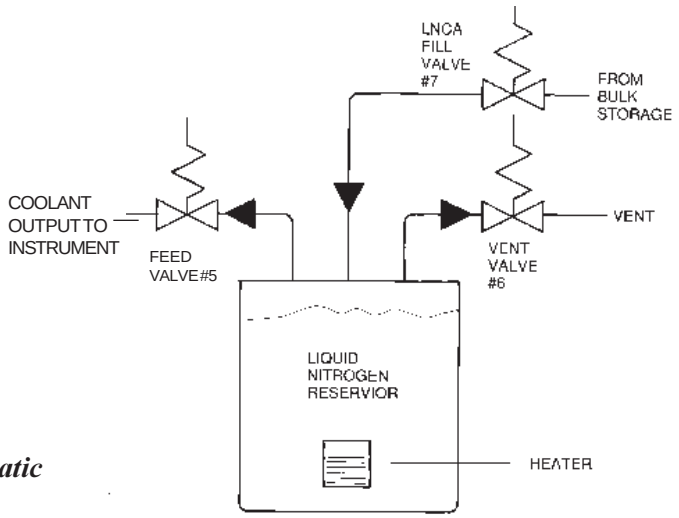

Figure 1.2
LNCA CE Schematic Operation

Description of Components

The following illustration shows the major parts of the TA Instruments LNCA CE. Refer to Table 1.4 for a description of these parts.

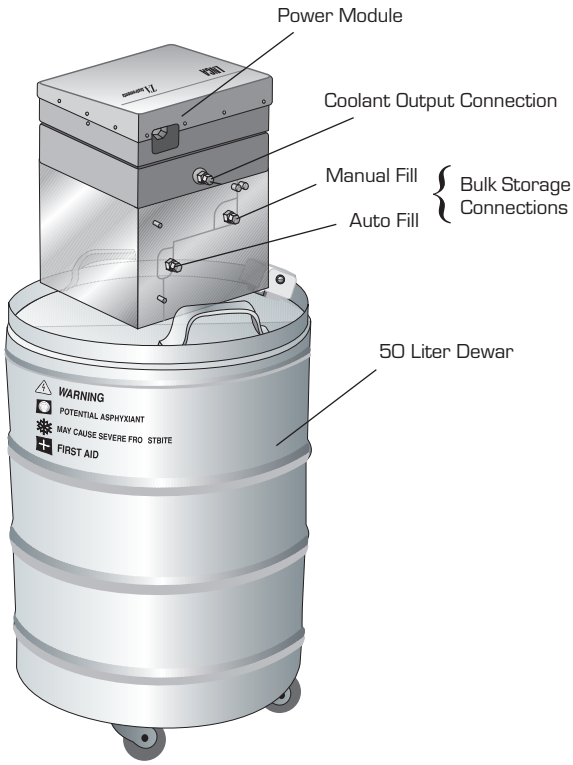

Figure 1.3 Major Parts of the LNCA CE

Table 1.4
LNCA CE Components

Item	Description
Power Module	Three-function IEC 320 power entry module containing power cord inlet, fuse, and on-off switch.
Manual Fill	Allows the LNCA CE to be refilled manually with connection of appropriate fitting.
Auto Fill	Automatically actuated solenoid valve located inside LNCA CE unit and activated to replenish LNCA CE coolant.
50 Liter Dewar	The thermally insulated storage vessel for liquid nitrogen.
Coolant Output Connection	Supplies a mixture of liquid and gaseous nitrogen to the test instrument.

Top Section of LNCA CE

Figure 1.4 illustrates the parts of the top section of the LNCA CE. Refer to Table 1.5 for a description of the individual parts.

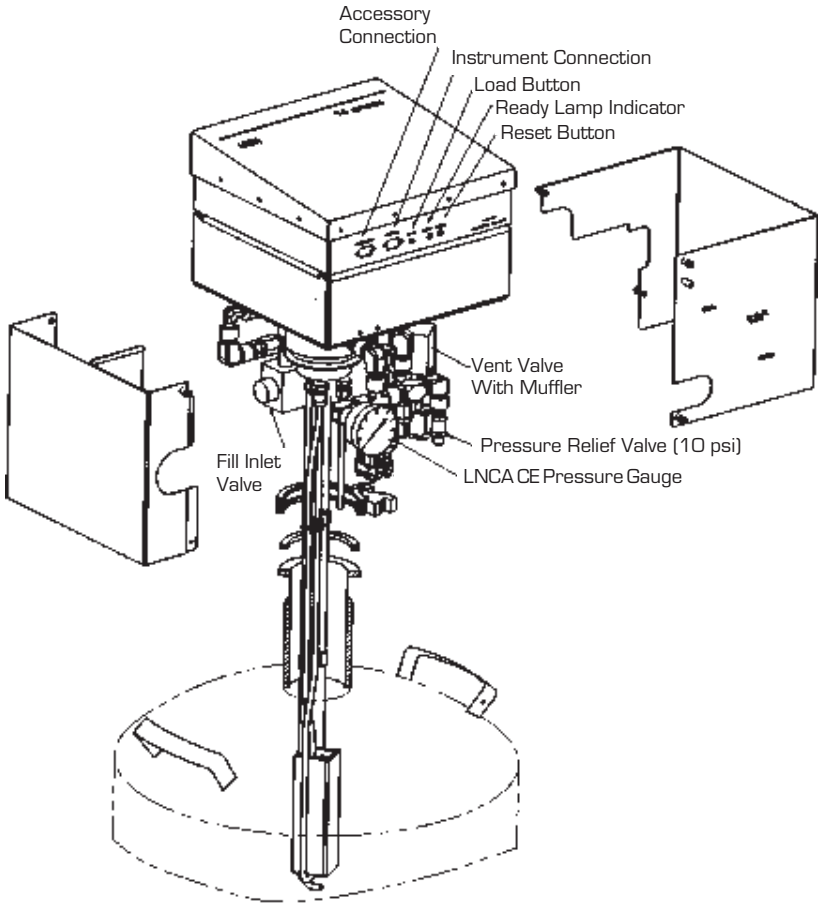

Figure 1.4 Top Section of the LNCA CE

Table 1.5
Parts of the LNCA CE's Top Section

Item	Description
Fill Inlet Valve	Solenoid valve that controls automatic refilling of LNCA CE from bulk storage.
Muffler	Located at the exit to the vent valve to reduce noise level during the refilling process.
LNCA CE Gauge	Pressure gauge located near the bypass valve to indicate LNCA CE pressure.
Pressure Relief Valve	Pressure relief valve that opens automatically to relieve pressure in the LNCA CE above 10 psi.
Bypass Valve	Valve that can be opened manually to refill the LNCA CE and to reduce the fill time.
Vent Valve	An automatically actuated solenoid valve that opens to relieve pressure when testing is complete and to allow refilling of the LNCA CE.

(table continued)

Table 1.5 (continued)

Item	Description
Ready Lamp Indicator	Ready lamp is illuminated once the power to the LNCA CE is turned on and the LNCA CE computer has successfully performed its self test. Flashing indicates error during confidence test.
Reset Button	An interrupt switch that clears and reboots the LNCA CE computer so that the LNCA CE need not be switched off.
LOAD Button	The LOAD button, when activated, puts the CPU in a load state. This is used when software changes are performed.
Accessory Port	Additional port for gang connecting other accessories.
Interface Cable (not shown)	RS-232 data transmission cable provides control signals from the instrument controller to the LNCA CE through the port labeled INSTRUMENT.

Chapter 2: Installing the LNCA CE

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 LNCA CE 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 LNCA CE.

If the LNCA CE 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 LNCA CE

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

WARNING

Read the safety precautions for handling cryogenic materials (located in the preface of this manual) before filling the LNCA CE. 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.

The following are general guidelines to use when installing the LNCA CE.

1. Check the clamp holding the LNCA CE Controls to the dewar. Sometimes, this clamp loosens during shipment. Access to the clamp is obtained by removing one or both of the lower covers.

2. The Autofill feature is connected to the LNCA CE the same way for all instrument installations.

3. The transfer tube connections from the LNCA CE to the instrument are the same, except that 90° elbow fittings have been supplied for some cooling accessory connections.

Choosing a Location

Because of the sensitivity of experiments using the LNCA CE, it is important to choose a location using the following guidelines. Your LNCA CE 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 (120 volts AC, 50 or 60 Hz, 15 amps). A step-up/down line transformer may be required if the unit is operated at a higher or lower line voltage.
- ... your TA Instruments thermal analysis controller.

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

General Installation Instructions

Installation of the LNCA CE with each type of instrument varies slightly. This section provides a general set of instructions that you can use to install the LNCA CE on any DSC instrument. For details on a particular type of instrument, refer to the appropriate section later in this chapter.

The LNCA CE is designed to be filled automatically from a *low pressure* (20 to 25 psi) bulk storage liquid nitrogen container. If you will not be using the Autofill feature, go directly to page 3-6 for manual filling instructions.

♦ ***CAUTION:***

If your liquid nitrogen source has more than 25 psi, then a pressure regulator must be added to ensure that no more than 25 psi is delivered to the LNCA CE. 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.

!WARNING

The accessory power switch *must* be off before connections are made.

To use the auto refill capability, follow these steps:

1. Arrange the low pressure bulk storage source physically close enough, within 180 cm (6 feet), to the LNCA CE so that the autofill tube can be easily connected between the source and the LNCA CE. Likewise the LNCA CE and the instrument need to be in close proximity to allow connection of the 180 cm (6 foot) transfer tube.

2. Adapting the LNCA CE for automatic refilling is very easy:

   a. Make sure the manual bypass valve at the front of the shroud is closed.

   b. Attach the transfer tube to the LNCA CE's Auto Fill fitting.

   c. 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. Connect the second transfer tube from the LNCA CE to the instrument and make sure the fittings are tight. The transfer tube connection at the instrument will vary according to the type of heat exchanger and instrument that you are using. See the appropriate section for the type of instrument you are using.

4. Make sure the power to the instrument is off and the power switch on the LNCA CE is in the off position.

5. Turn on the power to the LNCA CE and the instrument after connecting the interface cable.

Installling the Drain Valve

Ice and frost are created during normal use of the LNCA CE. The LNCA CE catch trough is designed to prevent water from dripping onto the floor and creating a potential hazard when the ice and frost melt.

A drain valve is supplied to permit occasional emptying of any water buildup from inside the condensation ring. To install the condensate drain valve, use a 5/8-inch wrench on the swage nut, and screw the elbow into the fitting until it is hand tight with the valve pointing down (see Figure 2.1).

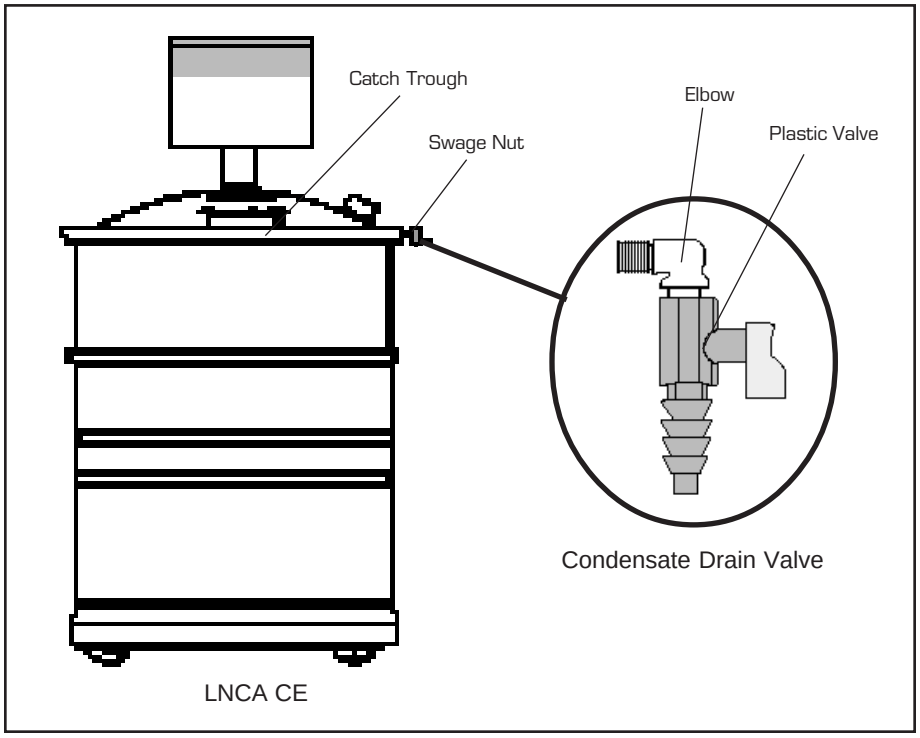

Figure 2.1
Drain Valve Installation

Water inside the condensation ring should 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 LNCA CE tank, causing liquid nitrogen to spill into the catch trough.**

Installing the LNCA CE on the DSC 2010 CE

The Liquid Nitrogen Cooling Accessory (LNCA CE) is used to achieve continuous, programmed sample cooling with the DSC 2010 CE Cell. With the LNCA CE installed, the temperature range of DSC experiments is –150°C to 725°C.

To operate the LNCA CE with the DSC Cell, you must first install the LNCA CE heat exchanger over the DSC Cell, and then connect the LNCA CE to the heat exchanger. Optional modification of the DSC 2010 CE cell results in permanent changes to the cell. Once modified, the DSC 2010 CE can no longer be used with the Refrigerated Cooling System. Furthermore, although the LNCA CE heat exchanger may be removed and the nickel cover replaced, it is recommended that once the LNCA CE heat exahanger is installed, you leave it attached to your DSC 2010 CE.

Installing the DSC Cell Heat Exchanger

To perform subambient experiments using the LNCA CE, you will need to install the DSC Cell Heat Exchanger and the DSC insulation tape, as follows:

Wear protective gloves when working with the insulation tape. Familiarize yourself with the MSDS for the insulation material as skin, eye, and respiratory irritation may occur, if you are exposed to the material.

Before you begin, make sure the cell has cooled to ambient temperature to avoid injury.

1. Turn the instrument POWER switch to the OFF (0) position, and unplug the instrument.

2. Remove the bell enclosure, O-ring seal, and cell cover from the DSC cell.

♦ ***CAUTION:*** **Make sure that the power to the 2010 CE instrument is OFF and the insturment is unplugged before installing the heat exchanger.**

3. Remove the three (3) retaining screws, then *carefully* remove the thermal radiation shield surrounding the heater. (see Figure 2.2).

♦ ***CAUTION:*** **Do not twist or apply excessive force to the radiation shield mounting plate when removing the shield. Damage to the furnace supports could result.**

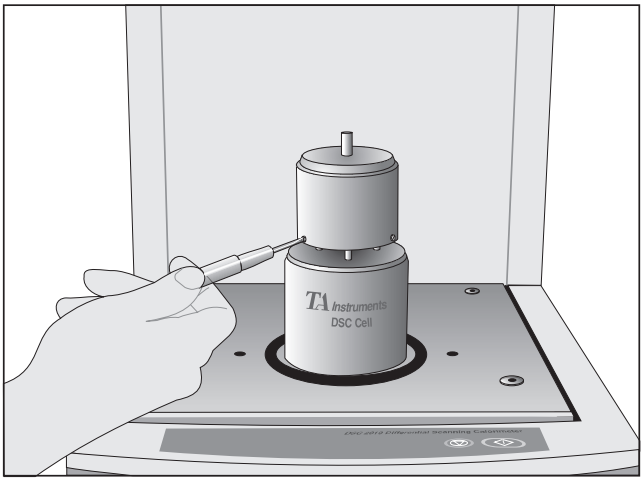

Figure 2.2 Removal of the Thermal Radiation Shield

4. Place the three gaskets onto the cell base plate as shown in Figure 2.3, aligning the holes in the gaskets over the two threaded holes in the base plate.

5. Thread the two hex-socket screws through the gaskets several turns, but do not tighten them all the way down at this point.

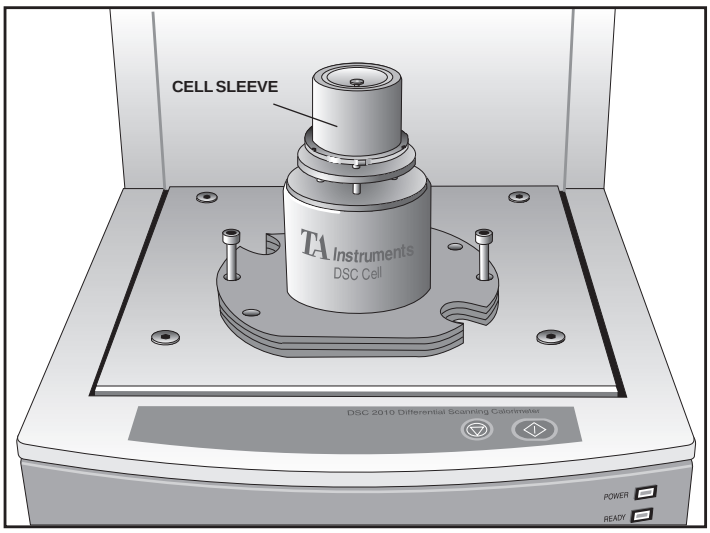

Figure 2.3 Position of the Gaskets

6. Remove the three (3) screws that secure the cell sleeve to the cell heater support.

7. Gently lift up on the sleeve, while applying small clockwise and counterclockwise twisting forces, to carefully remove the cell sleeve.

NOTE:

Take care when removing the cell sleeve. It is important to avoid deforming the cell support and causing the cement under the sleeve to fall off. However, small amounts of cement dust are normal and will not adversely affect performance. Cracks in the cement are also normal and occur from shrinkage during the curing process.

8. Install the insulation tape on the cell as directed in the following steps. The purpose of the insulation material is to obtain maximum performance from the DSC when it is used with the LNCA CE. The insulation ensures a uniform temperature around the cell, which lowers noise (μW) and flattens the baseline.

    a. Remove the backing strip from the insulation tape to expose the adhesive side.

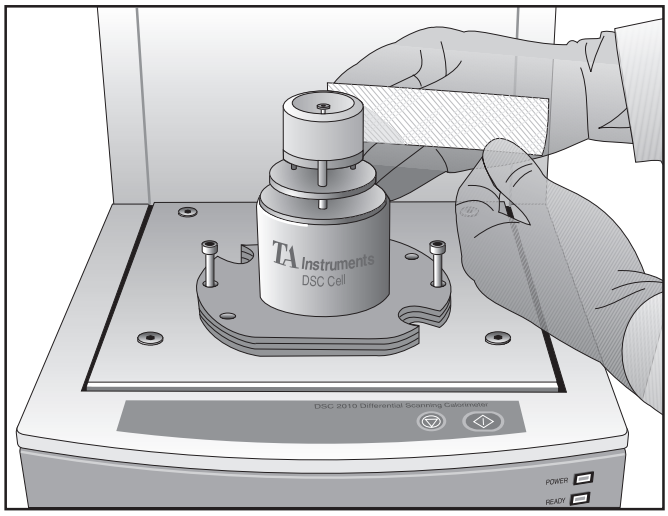

Figure 2.4 Installing the Insulation on the DSC Cell

    b. Position the insulation beginning at the back of the cell sleeve and allowing 3.2 mm (1/8 inch) to extend above the top of the cell (see Figure 2.4).

    c. Apply the insulation slowly and evenly, wrapping it around the cell and stretching it as you go. When you reach the midline on the back of the cell, where the ends of the insulation will meet, it is very important to form a butted insulation seam (with no overlap and minimal gapping.)

d. Use scissors to cut off any excess insulation at the seam to prevent overlap, then cut off any stray threads around the top of the cell.

e. Gently fold the insulation that extends above the cell inward toward the center of the cell. This is to prevent the heat exchanger from dislodging the insulation when it is installed.

9. Plug in the heat exchanger cable as shown in Figure 2.5.

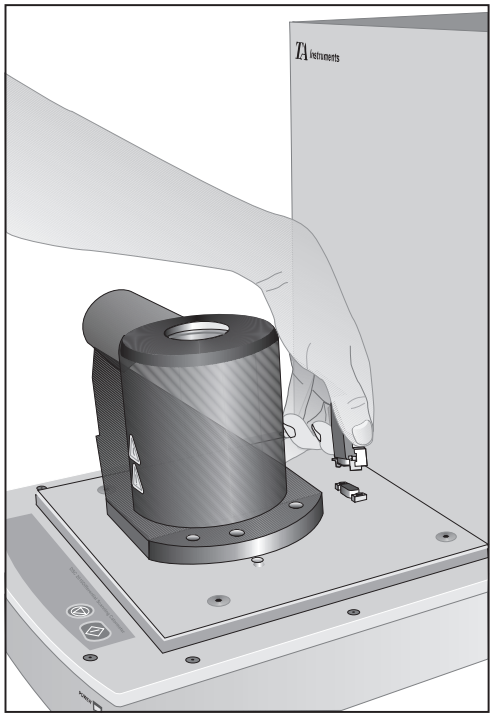

Figure 2.5 Plugging in the Heat Exchanger

10. Carefully lower the heat exchanger assembly down over the DSC cell with the gas inlet port facing toward the left side of the cell base (see Figure 2.6).

♦ ***CAUTION:***

The heat exchanger assembly contains coated fiberfax material. Excessive handling of this material could cause fiberfax particles to be emitted into the air. See the MSDS sheet for safety measures to be observed when fiberfax is used.

If the cell is twisted or canted, it can bind against the inside of the heat exchanger, causing periodic slippage during heating or cooling scans. This slippage can cause shifts in the DSC heat flow baseline.

The cell should be aligned with the heat exchanger so that the exchanger assembly slides smoothly up and down on the cell and sits squarely on the rubber gasket at the base.

11. Align the holes in the heat exchanger with the hex-socket screws in the gaskets. Once the heat exchanger is in place, rotate it clockwise into the locking position.

12. Tighten the hex-socket screws to secure the DSC Cell Heat Exchanger to the cell base plate (refer to Figure 2.6 on the next page). When bolted in place, the heat exchanger must not press against the outside of the cell furnace. With proper installation, it should be possible to move the cell *very slightly* in all lateral directions by pressing with a finger against the inner furnace wall.

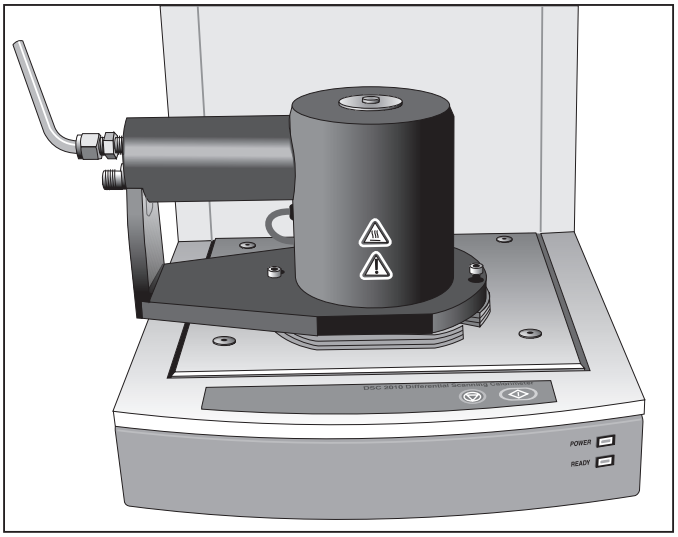

Figure 2.6 DSC Cell Heat Exchanger Assembly on the DSC 2010 CE

WARNING

Do not block the nitrogen gas outlet port on the heat exchanger. Avoid prolonged contact with the cold outlet gas stream.

13. Place the two new lids on the DSC cell heat exchanger.

NOTE:

When running subambient, use a dry nitrogen purge through the vacuum port (100–150 mL/min) to eliminate moisture buildup.

Before you can begin to use the cell, you must burn off the adhesive material used during installation of the insulation.

Burn-Off Procedure

1. Use a minimum of 50 cc/min of nitrogen purge in the Purge Port on the back of the cell base.

2. Remove all lids, except the silver lid, which covers the sample chamber of the cell.

NOTE: Use an exhaust system to carry away the fumes that are generated during the decomposition of the adhesive.

3. Run the following method:

   Equilibrate at 300°C
   Ramp 10°C/minute to 600°C
   Isothermal for 10 minutes.

4. Adjust the nitrogen purge to the desired flow rate.

5. Connect the LNCA CE transfer tube to the heat exchanger, following the instructions found beginning on page 2-18.

6. Calibrate the cell as described in the manual.

NOTE: Removal of heat exchanger will require a repeat of the insulation process.

Connecting Cable and Tubes

The new components installed in this section are shipped with the LNCA CE or come from the LNCA CE/DSC installation kit. Before beginning this procedure, install the DSC heat exchanger as described in the previous section.

!WARNING

Liquid nitrogen trapped in tubes and valves can cause dangerous pressure buildup when it vaporizes. Never use tubing or valve configurations that could trap liquid nitrogen in your LNCA CE system.

1. Connect the LNCA CE coolant transfer tube from the LNCA CE feed connection marked "COOLANT" to the stainless steel union on the heat exchanger end plate (Figure 2.7).

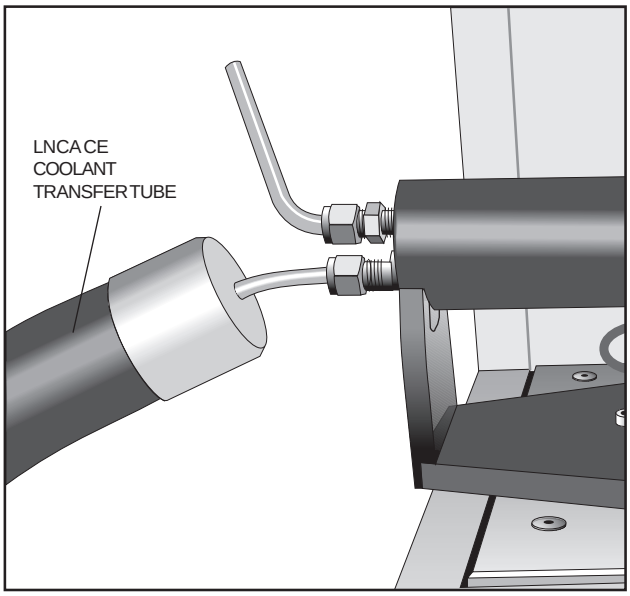

Figure 2.7 Installing the LNCA CE Coolant Transfer Tube

2. Install the short, black foam insulation tubing over the exposed stainless steel end of the LNCA CE coolant transfer tube. This tubing is slit so that you can install and remove it without disconnecting the LNCA CE coolant transfer tube.

3. Install the accumulator *(optional)* to collect excess coolant as follows:

   a. Unscrew the vent tube to remove it from the heat exchanger.

   b. Attach the elbow connector to the vent tube by pushing the end of the vent tube into the connector. Then tighten the fitting to hold securely.

   c. Screw the accumulator onto the threaded part of the elbow fitting. You can position the opening to the accumulator as desired, it does not need to be tightly attached (see Figure 2.8).

   d. Screw the vent tube back into the heat exchanger, positioning it so that it is horizontal.

NOTE: When positioning the accumulator, make sure that the vent opening does not point toward you, because cold gas will be escaping.

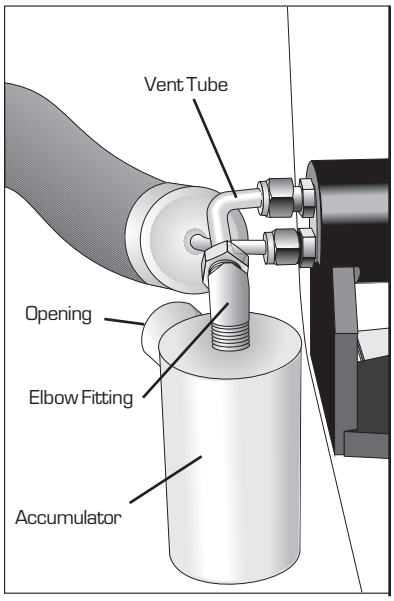

Figure 2.8 Installing the Accumulator

4. Make sure the power to the instrument is off, the instrument is unplugged, and the LNCA CE power switch is in the OFF (0) position.

5. Connect the LNCA CE interface cable from the LNCA CE connector port marked "INSTRUMENT" to the "LNCA" connector on the back of the instrument. Tighten the cable thumbscrews.

6. Plug in the LNCA CE power cord, and turn on the LNCA CE power switch. The instrument is now ready for operation.

Installing the LNCA CE on the DSC 2920 CE

The Liquid Nitrogen Cooling Accessory (LNCA CE) is used to achieve continuous, programmed sample cooling with the standard and dual sample DSC cells. With the LNCA CE installed, the temperature range of DSC experiments is –150°C to 725°C.

To operate the LNCA CE with a DSC cell, you must first install the DSC heat exchanger over the DSC Cell, and then connect the LNCA CE to the DSC heat exchanger. The heat exchanger comes in the LNCA CE /DSC installation kit, which also contains some additional parts and installation instructions.

Installing the DSC Heat Exchanger

Follow these steps to mount the heat exchanger and prepare your DSC Cell for subambient experiments using the LNCA CE . All new components installed in this section come in the LNCA CE /DSC installation kit.

1. Remove the bell enclosure, O-ring seal, and cell cover from the DSC Cell (see Figure 2.9).

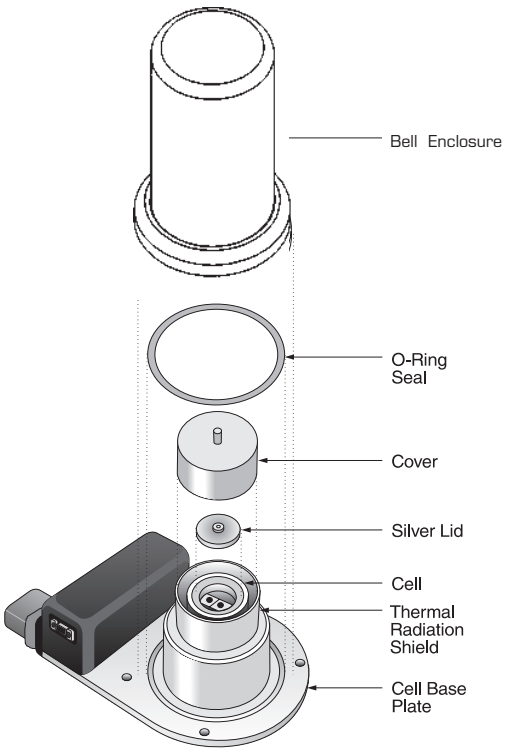

Figure 2.9 Disassembling the DSC Cell

WARNING

Make sure the cell is at ambient temperature before touching it, to avoid being burned.

2. Check that the instrument POWER switch is off, and unplug the instrument.

♦ ***CAUTION:***

Make sure that the power to the 2920 CE instrument is OFF, and the instument unplugged, before you perform these procedures.

NOTE:

If you remove the thumbscrews on the DSC 2920 CE, the power to the cell is automatically disconnected. (This does not occur with the DSC 2010 CE).

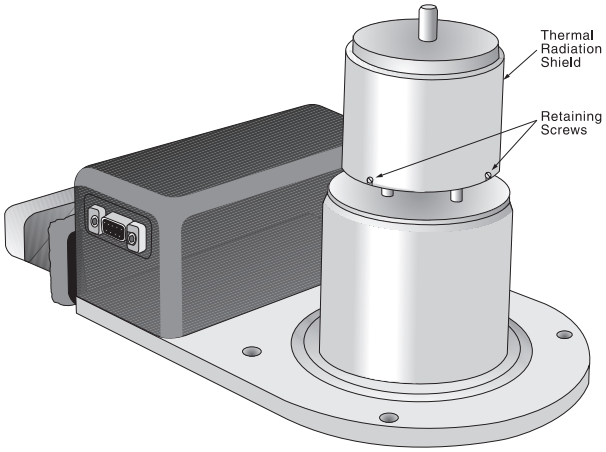

Figure 2.10
DSC Standard Cell

3. Loosen the three retaining screws on the thermal radiation shield, and remove it by pulling it straight up. The slots in the shield allow easy removal (refer to Figure 2.10 for location of screws).

NOTE:

For better performance, you can install the optional DSC insulation tape on the 2920 cell at this point. Follow the instructions given for the DSC 2010 CE, found on pages 2-13 through 2-14, steps 1 through 8, before going on to step 4 on page 2-24.

4. Fit the three new gaskets onto the cell base plate (see Figure 2.11), aligning the holes in the gaskets over the two thumbscrews.

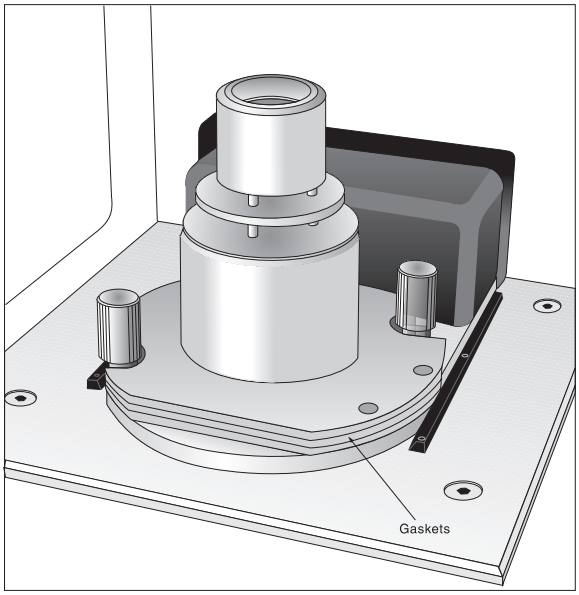

Figure 2.11 Installing the New Slotted Gaskets

5. Align the arm of the heat exchanger to the left, and carefully lower it down over the thumbscrews, taking care not to hit the cell. See Figure 2.12.

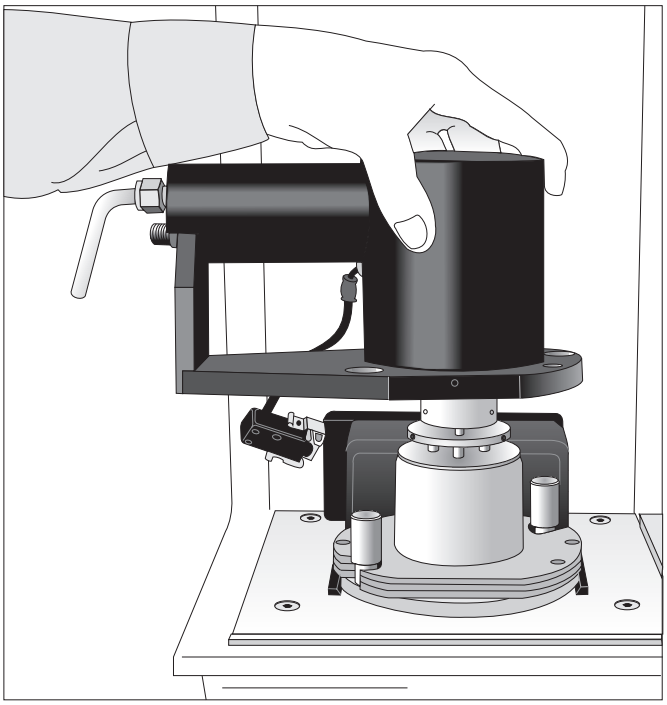

Figure 2.12 Installing the Heat Exchanger

WARNING

The heat exchanger assembly contains coated fiberfax material. Excessive handling of this material could cause fiberfax particles to be emitted into the air. See the MSDS sheet for safety measures to be observed when fiberfax is used.

If the cell is canted it can bind against the inside of the heat exchanger, causing periodic slippage during heating or cooling scans.This slippage can cause shifts in the DSC heat flow baseline.

The cell should be aligned with the heat exchanger so that the exchanger assembly slides smoothly up and down on the cell and sits squarely on the rubber gasket at the base.

6. Screw the two large thumbscrews into their positions in the heat exchanger (see Figure 2.13).

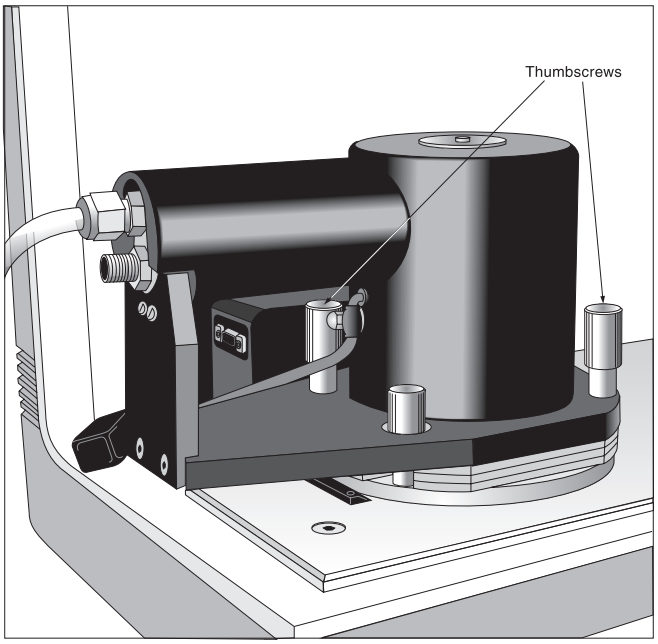

Figure 2.13 Installing the Thumbscrews on the Heat Exchanger

7. Plug in the cable from the heat exchanger (see Figure 2.14 for the cable setup on the DSC 2920 CE), making sure that the spring clip retainers are fully engaged.

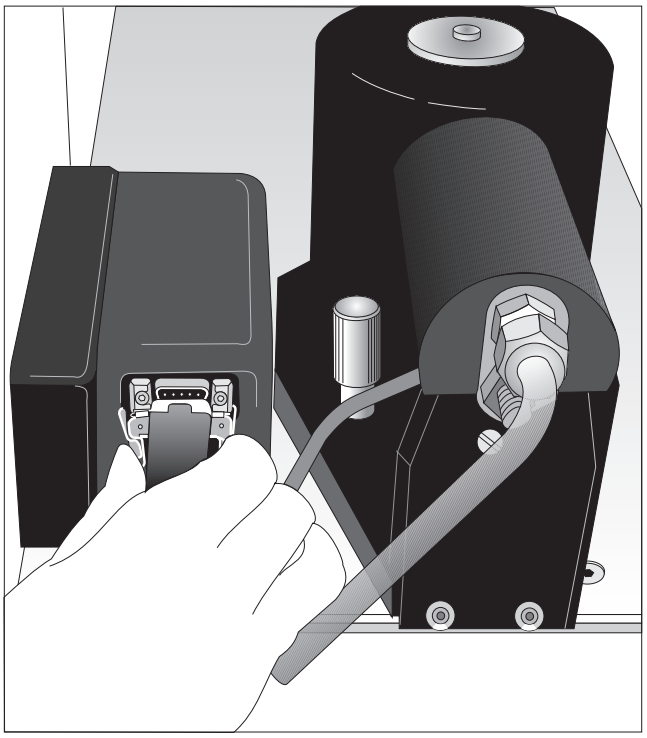

Figure 2.14 Plugging in the Heat Exchanger (DSC 2920 shown)

NOTE:

If you installed DSC insulation tape on your 2920 cell, you must perform the "Burn-Off Procedure" found on page 2-17 before using the instrument. This procedure burns off the adhesive material used during the installation of the insulation tape.

Connecting Cable and Tubes for Cooling

The new components installed in this section are shipped with the LNCA CE or come from the LNCA CE/DSC installation kit.

!WARNING

Liquid nitrogen trapped in tubes and valves can cause dangerous pressure buildup when it vaporizes. Never use tubing or valve configurations that could trap liquid nitrogen in your LNCA CE system.

1. Install the heat exchanger on the DSC 2920 CE instrument as described previously.

2. Connect the LNCA CE coolant transfer tube from the LNCA CE feed valve marked "COOLANT" to the stainless steel union on the heat exchanger end plate (see Figure 2.15).

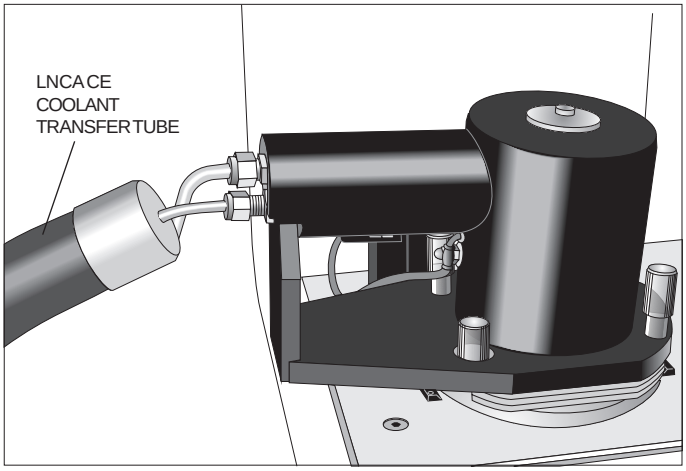

Figure 2.15
Installing the LNCA CE Coolant Transfer Tube

3. Install the accumulator *(optional)* to collect excess liquid nitrogen as follows:

   a. Unscrew the vent tube to remove it from the heat exchanger.

   b. Attach the elbow connector to the vent tube by pushing the end of the vent tube into the connector. Then tighten the fitting to hold securely.

   c. Screw the accumulator onto the threaded part of the elbow fitting. You can position the opening to the accumulator as desired; it does not need to be tightly attached (see Figure 2.16).

   d. Screw the vent tube back into the heat exchanger, positioning it so that it is horizontal.

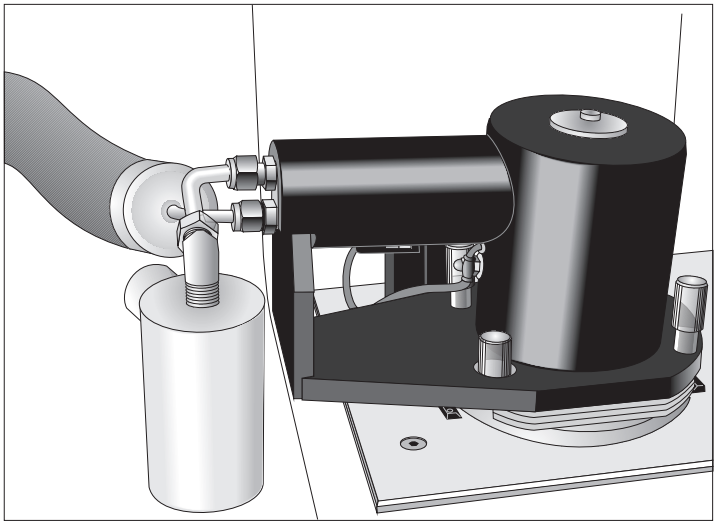

Figure 2.16 Installing the Accumulator

NOTE: When positioning the accumulator, make sure that the vent opening does not point toward you, because cold gas will be escaping.

4. Make sure the power to the instrument is off and the LNCA CE power switch is in the OFF (0) position.

5. Connect the LNCA CE interface cable from the LNCA CE connector port marked "INSTRUMENT" to the "LNCA CE" connector on the back of the instrument. Tighten the thumbscrews on the interface cable.

6. Place the silver lid on the cell and the two new lids on the DSC heat exchanger.

NOTE: When running subambient, use a dry nitrogen purge through the vacuum port (100–150 mL/min) to eliminate moisture buildup inside the cell.

The software used to run the LNCA CE has already been loaded at TA Instruments. However, you may need to update that software with a new version. Follow the instructions in the next section to update the GCA program.

After the LNCA CE has been completely installed, and the software updated if necessary, you can turn to the next chapter for the instructions needed to fill the accessory with liquid nitrogen.

Updating the Software

If you need to update the software with a new version, follow these instructions to connect the LNCA CE to the controller and to run the TA Instruments Accessory Loader program:

NOTE: The windows appearing in this section reflect the *Thermal Solutions/Advantage* software. If you are using the OS/2 controller software, the windows will appear slightly different but the procedure is still basically the same.

1. Disconnect the RS-232 interface cable from the instrument.

2. Connect the RS-232 interface cable from your LNCA CE to your controller. (You may need to disconnect both ends of the RS-232 cable, then reverse the ends so that the proper gender connectors match.)

NOTE: You can connect the RS-232 cable from the controller to either the INSTRUMENT or ACCESSORY connection on the LNCA CE.

3. Start the Accessory Loader program from the **Start/Programs** menu. The window shown in the figure on the next page is displayed.

4. Select the **Start** button to begin loading the program. The program will check to make sure a TA Instruments accessory is connected to a communications (COM) port on the controller.

The button will change to read **"Pause,"** to allow you to halt the loading process, if desired.

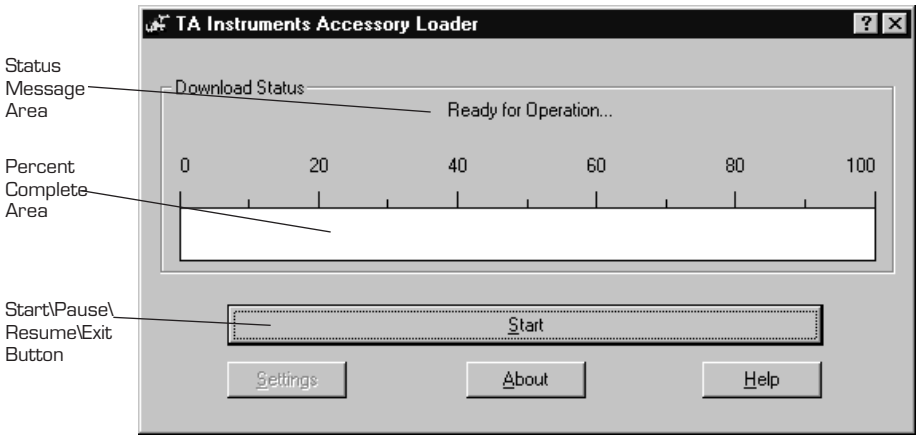

Figure 2.17
Accessory Loader Window

Refer to Table 2.1, on the next page, for a description of the parts of the Main window.

Table 2.1
Parts of the
Main Window

Part	Description
Status Message Area	As the program is loading, this area will display text describing the action in progress.
Percent Complete Area	A status bar is filled in from left to right as the program loads, providing a visual method to track the percent completion of the procedure.
Start/ Pause/ Resume/ Exit Button	This main control button changes depending on the current point in the loading process. "Begin" is used to start the loading process. "Suspend" is used to halt the program and place it on hold. "Resume" is used to restart the program after it was suspended. "Exit" is used, after the program has completely loaded the software, to end the program.

If no LNCA is found, you can change the program settings, if needed, by following the instructions found in the section entitled "Changing the Settings" on page 2-37.

If the LNCA is located successfully, the following message is displayed.

Figure 2.18
Load Button Message

3. Locate the recessed Load button on the side of the LNCA, shown in the figure below.

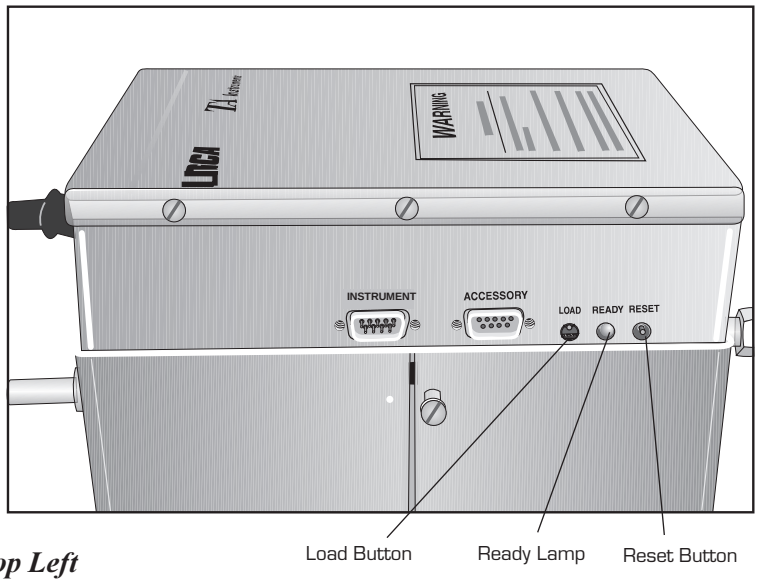

Figure 2.19
LNCA CE Top Left Section

4. Using a blunt-end tool, such as a pen tip, depress the Load button. The Ready lamp should go off to indicate that you have successfully placed the accessory into the "load" state.

5. Click the **Resume** button on the **Accessory Loader** window.

NOTE: You can halt and cancel the download process by selecting the **Cancel** button.

The window shown below is displayed.

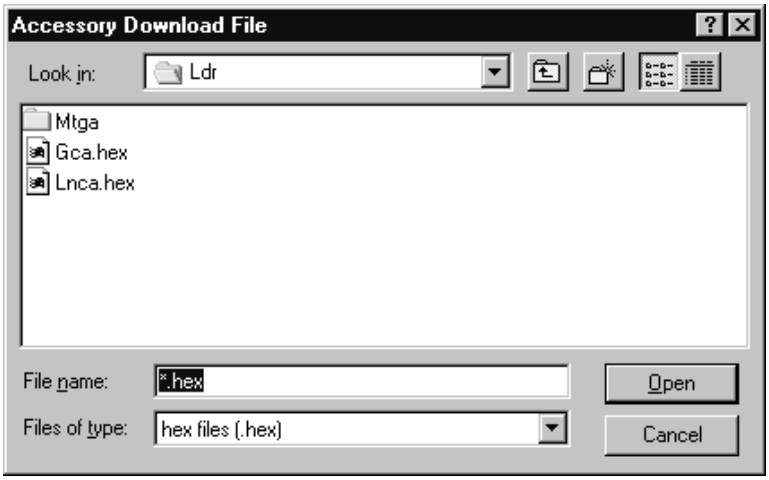

Figure 2.20
Select Loader File Window

6. Select the proper HEX file (Lnca.hex), then select **OK**.

The program will read the file and send the appropriate information to the accessory. The Download Status area will reflect the status of the procedure. Then the message shown in the figure below will be displayed.

NOTE:

You can pause and resume the operation at any time during the downloading procedure using the appropriate button. You can also end the procedure by closing the window.

Figure 2.21
Reset Accessory Message

7. After the software has been successfully loaded, press the Reset button on the side of the LNCA CE top section, see Figure 2.19 for its location. This will retrieve the calibration information used for the LNCA CE and reset the accessory. (You may need to turn the power to the LNCA CE off, and then on again, if the Reset is not successful.)

   The status message area on the **Main** window will display "Software Download Successful!"

8. Select the **Exit** button.

Changing the Settings

The Liquid Nitrogen Cooling Accessory has been set up to run using the default settings used in the Accessory Loader program. Therefore, we recommend that you keep the settings as shown in the figure below.

To access the **Communications Settings** window, select the **Settings** button on the **Accessory Loader** window.

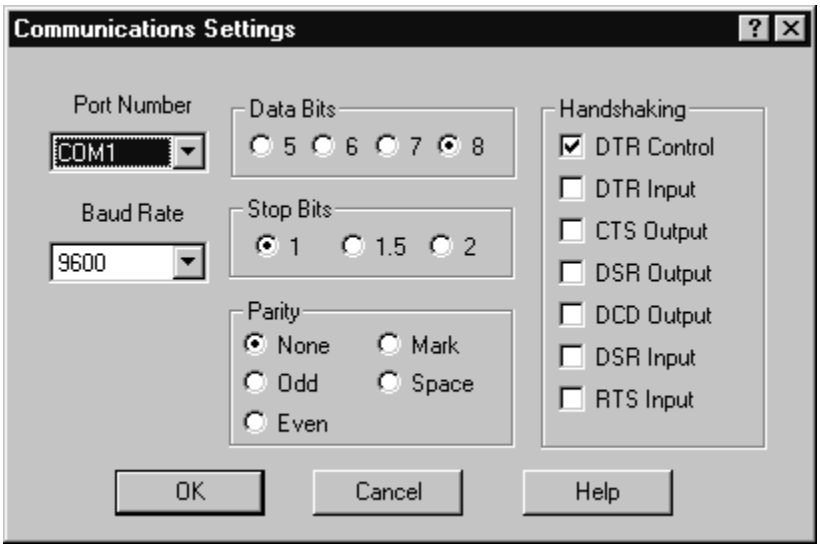

Figure 2.22
Communications Settings Window

The table on the following page provides a brief explanation of the settings shown.

Table 2.2 Communications Settings

Setting	Description
Port Number	The communications port used to connect an accessory to your controller. Select either COM 1 or COM 2 depending upon your system setup. *Default setting = COM1*
Data Bits	Number of bits per character. *Default setting = 8.*
Handshaking	The required sequence of signals for communication between the LNCA and the controller. *Default setting = DTR Control.*
Baud Rate	The transmission rate for data signals. *Default setting = 9600*
Stop Bits	The last element of a character that signals the end of the character. *Default setting = 1.*

(table continued)

Table 2.2
Accessory Loader
Settings (cont'd)

Setting	Description
Parity	A bit, transmitted before the stop bit, that is used to check for errors in communications. *Default setting = None.*

Chapter 3: Filling and Operating the LNCA CE

Autofilling the LNCA CE

"Autofilling" refers to the automatic refilling of the LNCA CE from the bulk storage tank between runs. This section tells you how to set up the LNCA CE and the connected instrument to allow autofilling. Turn to the procedure appropriate to the instrument you are currently using.

NOTE: When liquid nitrogen enters the dewar for the first time, rapid boil-off of the liquid nitrogen occurs. On occasion, liquid nitrogen may exit the vent, causing the fill sensor to prematurely indicate a full tank. If this occurs during the initial filling, wait a few minutes, and then restart the fill process.

The LNCA CE can be used to achieve programmed cooling of the DSC 2010 CE or the DSC 2920 CE and their accessories.

The 2000 series instruments can automatically control the pressure in the LNCA CE supplying liquid and gaseous nitrogen to the instrument.

This chapter explains how to fill and use the LNCA CE now that it is connected.

For information on running subambient experiments, refer to the appropriate chapter in your instrument's operator's manual.

Initial Filling of the LNCA CE

The LNCA CE must be filled before cooling experiments can be performed on 2000 series instruments. Follow the instructions in this section to fill the LNCA CE to be used with a 2000 series instrument for the first time.

TA Instruments RMX-Based Software

1. Set the method-end conditions to select autofilling at the end of a method.

2. Create, load, and run the following method to initiate filling the dewar.

   Jump to 0°C
   Isothermal for .2 minutes

The autofill will shut off when the dewar is full or when the LNCA CE tank pressure is below 1 psi for more than 1 minute, usually because the bulk storage tank is empty.

TA Instruments Thermal Solutions/Advantage Software

Select **Control/Auto Fill** on the Instrument Control pop-up **Main Menu**. The LNCA CE will be filled automatically.

The autofill will shut off when the dewar is full, the bulk storage tank is empty, or the LNCA CE tank pressure is below 1 psi for more than 1 minute.

Refilling the LNCA CE After an Experiment

To automatically refill the LNCA CE with liquid nitrogen after an experiment on a 2000 series instrument is completed, you simply set the method-end conditions to select autofilling at the end of a method. This applies to both the RMX-based software and *Thermal Solutions/Advantage* software.

Manually Filling the LNCA CE

The LNCA CE is designed so that it can be filled *manually* as well as automatically. The automatic filling procedure is discussed in the previous section. These instructions explain the method of manually filling the LNCA CE. The Manual Fill mode should be used only if a bulk storage reservoir cannot be placed close to the LNCA CE.

Follow the directions in this section to fill the LNCA CE.

1. Turn off the power to the instrument.

2. Disconnect the LNCA CE coolant transfer tube from the LNCA CE coolant output connection.

3. Disconnect the interface cable and power cord.

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

5. Roll the LNCA CE, if necessary, to the location of the bulk storage source.

6. Connect the coolant transfer tube to the LNCA CE bulk storage supply, using the union and adapter fitting supplied in the accessory kit.

7. Open the bypass valve on the front of the LNCA CE, to enable the gas to escape from the LNCA CE and allow it to fill more rapidly.

8. Uncap the Manual Fill fitting of the LNCA CE, and connect the other end of the feed tube to this fitting.

9. Open the valve on the bulk storage source to begin filling the LNCA CE.

NOTE: Cold gas will escape from the LNCA CE bypass valve and the vent valve during the filling process. The fill process normally takes 30–40 minutes.

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

10. Fill the LNCA CE until spurts of liquid escape from either the vent valve or the bypass valve.

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

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

13. Disconnect the LNCA CE coolant transfer tube from the LNCA CE manual fitting fill connection.

14. Re-cap the Manual Fill fitting.

15. Close the LNCA CE Bypass valve, and disconnect the LNCA CE coolant transfer tube from the nitrogen supply tank.

16. Reposition the LNCA CE near the analysis instrument, and reconnect the following:

    - Interface Cable
    - Coolant Transfer Tube to the LNCA CE coolant output fitting
    - LNCA CE Power Cord.

Operating the LNCA CE

The Liquid Nitrogen Cooling Accessory (LNCA CE) is a self-contained dewar flask that is connected to the different thermal analyzer instruments by an insulated coolant transfer tube and an electrical interface cable.

Operation of the LNCA CE with the 2000 series DSC instruments first requires the installation of a heat exchanger, as directed in Chapter 2 of this manual.

After the heat exchanger has been installed, fill the LNCA CE as directed earlier in this chapter. To operate the LNCA CE with the DSC 2010 CE or DSC 2920 CE, create a cooling method, if desired, and simply set the method-end conditions to autofill the LNCA CE at the end of your method.

Appendix A:
Ordering Information

Parts Ordering Information

Service should only be performed by qualified service personnel. Please contact TA Instruments at one of the offices listed on page A-5 for service or replacement parts. To ensure that you receive the correct part for your unit, be sure to include the part number, description, instrument type, model number, and serial number.

Table A.1
LNCA Parts List

Part No.	Description
891050.901	Board, Main PC, LNCA Auto (A1)
270712.001	Cable, RS232
205225.037	Fuse
890035.901	Power Cord
264064.001	Gasket, Neoprene Tank, 3" Dia., LNCA
270172.001	Gauge, LNCA Tank Pressure, 0-30 PSI
281056.001	Heater Strip, LNCA
991041.901	Jet, Modified Tee, LNCA
891209.901	LNCA/CE Auto Fill Complete

(table continues)

Table A.1
LNCA Parts List
(Cont'd)

Part No.	Description
891300.001	Manual, Instruction, LNCA Auto
900816.901	Kit, Insulation, Adhesive-Backed
991286.903	Kit, Liquid Sensor—Upgrade
281072.002	Switch, Thermostatic Snap Disc, LNCA
991075.901	Tank, LNCA Replacement
991078.001	Tube, Transfer
891078.901	Valve, Solenoid, Coolant Supply, LNCA Auto (L4)
891075.901	Valve, Solenoid, Autofill, LNCA Auto (L3)
991284.901	Vent, LNCA Auto (L2)

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109 Lukens Drive
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Fax: 1-302-427-4001

HELPLINE—U.S.A.
For technical assistance with current or potential thermal analysis applications, please call the Thermal Analysis Help Desk at 1-302-427-4070.

SERVICE—U.S.A.
For instrument service and repairs, please call 1-302-427-4050.

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