

## **OPERATING INSTRUCTIONS**

### **LIQUID HELIUM DEWAR**

(VAPOR SHIELDED)

#### **1. DEWAR VACUUM**

The dewar has been shipped from the factory with the dewar insulating space evacuated. Initial or regular pumping of the dewar vacuum is not required. A vacuum maintenance system has been installed in the dewar which means that the dewar does not require pumping.

#### **2. PRECOOL CRYOSTAT**

Before introducing any liquid cryogen into the dewar, check carefully for any water in the helium reservoir. This water could have been introduced into the dewar via condensation on the walls during the previous 'run' (use). If not removed, this water will freeze upon cooling and could cause severe damage to the dewar and insert.

Insert a tube into the liquid helium reservoir.

*(A thin wall stainless steel tube, 0.375 in O.D. is recommended.*

*Grinding an angle on one end of the tube will prevent the tube from plugging if it reaches the bottom.)*

This tube should terminate close to the bottom of the helium reservoir.

Attach the tube to a supply of liquid nitrogen.

Open a dewar vent port. Fill the liquid helium reservoir with liquid nitrogen.

Allow time for the liquid nitrogen to cool the system. Several hours is recommended

and waiting overnight can be beneficial.

A short precool time can result in an initial higher consumption of liquid helium.

Note: A good precool substantially reduces the consumption of liquid helium used to fill the dewar and provides for lower LHe consumption rates immediately after transfer.

Helpful notes: (i). An economical heat gun, sold in most department stores, is ideal for removing extra frost formations.

### **3. `BLOW OUT' THE LIQUID NITROGEN FROM THE HELIUM RESERVOIR**

Remove the liquid nitrogen from the liquid helium reservoir.

Insert and seal a tube through a reservoir access port.

The tube should reach to **THE BOTTOM OF THE LIQUID HELIUM RESERVOIR.**

*(A thin wall stainless steel tube, 0.375 in O.D. is recommended.*

*Grinding an angle on one end of the tube will prevent the tube from getting blocked if it reaches the bottom.)*

Seal the tube so that a small pressure can be applied above the liquid nitrogen in the reservoir.

Attach a helium gas hose to a helium reservoir port or vent.

Pressurize the reservoir using a gas cylinder containing helium.

The liquid nitrogen will be `blown out' of the inserted tube.

**Important Note:** Try to assure that all the liquid nitrogen is removed.

Remove the tube and re-insert with the flat end into the dewar. Re-pressurize and 'blow' the remaining skim of LN2 up the tube. Move the tube up and down a little to make sure the tube is not blocked and all the the LN2 is removed.

Leave the reservoir back filled with helium gas. Install the helium reservoir pressure safety relief. All other helium ports should be plugged or stoppered closed. **Make sure that a positive pressure relief or check valve is used whenever the helium reservoir is sealed.**

Helpful note: Put a small angle on the end of your 'blow out' tube; this will prevent the common error of having a square end against the bottom of the reservoir which can prevent the removal of liquid nitrogen. In order to check that all the liquid nitrogen is removed, you can withdraw the 'blow out' tube and re-insert it so that the opposite flat end goes into the dewar.

#### **4. TRANSFER LIQUID HELIUM**

Make sure that all the liquid nitrogen used for precooling has been removed from the helium reservoir.

Note that ALL cold surfaces should be in contact with a cryogen, helium gas or vacuum **only - NEVER AIR!**

To transfer liquid helium, use a vacuum insulated transfer line.

For an **initial** fill, the transfer line 'leg' (downstream insert tube) should reach to the bottom of the liquid helium reservoir. The liquid helium entry point into the dewar should be below the insert and at the bottom of the reservoir; this will allow using the enthalpy of the helium gas in addition to the latent heat of vaporization of the liquid. (Note: For refills, the most efficient position for the transferring helium entry is above the existing liquid helium level. For initial fills [no liquid helium in the dewar], the most efficient position for the transferring helium entry is at the bottom of the reservoir.)

The transfer should start at a steady slow rate in order to make full use of the cooling power of the cold helium vapor, as it rises and escapes through the helium vent tube(s). In general, the 'push' pressure in the **storage** dewar should be less than one psig. After the liquid helium starts to collect, the transfer rate can be increased. Monitor the helium level using a level indicator. The amount of liquid helium needed to fill the dewar will depend on the size of the reservoir, the insert type, mass and materials installed and the efficiency of the transfer. In general, about five liquid helium liters, plus one liter per five pounds of inserted mass is required to cool the reservoir from 77 to 4.2 K. This, along with the overall capacity of the helium reservoir, should offer a rough figure for the amount of liquid helium required for initial cooldown.

Fill to your desired volume of liquid helium. Remember: For initial fill, most efficient transfer will occur if transfer line reaches the bottom of the reservoir. For refill, the transfer line should be inserted just above the liquid helium level.

Helpful notes:

Remember to allow the helium gas to vent freely during liquid helium transfer.

Set the liquid level monitor to 'continuous' mode during LHe transfer. Remember to set the LHe level monitor to 'Sample & Hold' after transfer is complete.

#### 5. INSTALL PRESSURE RELIEFS (Check Valves)

After the completion of the liquid helium transfer, **always install a safety pressure relief onto the helium reservoir**. The relief will maintain a positive pressure in the liquid helium reservoir preventing ice and contamination while protecting against over pressure. All other helium ports should be stoppered closed or manifolded and vented through the same pressure relief.

For dewars with multiple vent ports, all venting ports should be manifolded together and vented through one positive pressure relief. You may need to adjust the flow rate through each access port individually to achieve maximum efficiency. *(Usually the majority of the flow should pass through the neck to cool the vapor shields which are thermally anchored to the neck. Smaller amounts of gas can be used to intercept heat flow in other auxiliary access ports.)*

**OPERATE ONLY WITH THE SAFETY PRESSURE RELIEF IN PLACE; DO NOT REPLACE WITH A BLANK.**

#### 6. OPERATION BELOW 4.2 K (Pumped Bath)

Attach a mechanical pump to a vent port and pump on the liquid helium with a

mechanical pump.

WARNING - BE CAREFUL, REMOVAL OF THE POSITIVE PRESSURE ABOVE THE LIQUID HELIUM WILL RESULT IN A QUICK EVOLUTION OF COLD GAS. AVOID CONTACT!!! The roughing (mechanical) pump will reduce the boiling point of the liquid, bringing the system to the lowest possible temperature. Notes: (i). Due to vapor droplets, density changes, internal liquid cooling through self boiling, etc., it is normal to consume approximately 50% of the liquid helium volume before reaching terminal temperature.

(ii). Pumping on the helium reservoir should only be done with the dewar insulating vacuum intact. If there is no insulating vacuum in the dewar, check with the factory (Tel: (603) 893-2060) before pumping on the helium reservoir. In some of the dewar designs, the helium reservoir can implode under a reverse pressure of one atmosphere.

## 7. SHUT DOWN

- (i). Turn off all power to any sample mount(s) or inserts.
- (ii). Maintain the positive pressure regulator on the helium reservoir.
- (iii). Allow the system time to return to room temperature.

## GENERAL NOTES AND SAFETY PRECAUTIONS

1. If the dewar ever exhibits vacuum degradation, usually indicated by a significant increase in LHe consumption or a fully sweating (water condensing on the outside), consult the factory for correction.
2. Before cooling, visually assure that no water [condensed from the previous use] exists at the bottom of the cryogen reservoir. A large amount of ice formed at the bottom of the reservoir during prior use could damage the system.
3. Radiation baffles should be used in the neck of the dewar. Make sure the neck baffle allows the gas flow to divert to near the fiberglass neck for efficient cooling. All inserts installed into the liquid helium reservoir must have radiation baffles to prevent high liquid helium losses. Sample insert should properly baffle the liquid helium from room temperature radiative heat load. (Note: copper baffles can be used; also, a simple baffle can be made from most closed cell foams.)
4. Avoid contact with the cold gases.
5. The insulating vacuum space of the dewar is protected from over pressure by a disk o-ring seal on the 'head' of the dewar body. **DO NOT REMOVE!** Makes sure that the vent path for the relief is never restricted.
6. Install and maintain a safety pressure reliefs/check valve on the helium

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reservoir. The relief will maintain a positive pressure in the liquid helium reservoir preventing ice and contamination while protecting against over pressure.

7. Follow standard and proper cryogenic procedures. If you have any questions, we, at CRYO Industries, are only a phone call away and would be happy to answer any questions - (603) 621-9957 or FAX (603) 621-9960.