OPERATION INSTRUCTIONS

VARIABLE TEMPERATURE INSERT CRYOSTAT WITH SUPERCONDUCTING MAGNET

Introduction

Sample in vapor – low temperature inserts mount directly into transport (storage) or superconducting magnet research dewars. Samples are cooled by insertion into dynamic flowing helium gas entering the sample tube from pressure differential impedance. Liquid helium is drawn from the reservoir into the impedance. The liquid enters into the sample tube at reduced pressure and temperature.

Samples are top loading and can be quickly changed while operating. The combination of a sub lambda insert, superconducting magnet and a standard transport (storage) or vapor shielded dewar is a truly versatile and portable high precision laboratory tool.

Principle of Operation

Liquid helium is drawn from the dewar reservoir by pumping on the sample tube. The liquid enters into the sample tube at reduced pressure and temperature. The temperature (boiling point) of the liquid helium at the reduced pressure is sufficient low to allow operation of the QHR standard, typically between 1.2 – 1.3 K! The helium gas exits the sample tube and passes through the external vacuum pump.

Preparation

EVACUATE THE INSERT INSULATING VACUUM.

Pump the insulating vacuum of the insert while it is at room temperature. A high vacuum turbo type pump is recommended. If a high vacuum system is not accessible, a mechanical `roughing' pump will suffice. Pump the insulating vacuum with a high vacuum type pumping system **each** time the insert is cycled back to room temperature.

Never pump on the insert insulating vacuum with a mechanical (roughing) pump while it is inserted in the ‘cold’ storage dewar; mechanical pumps do not have low enough base pressure to pump against the cold cryopumping temperatures in the insert when cold. Plus, doing so may cause oil to backstream into the insert, if using an oil based pump. Pumping on the insulating vacuum space while the insert is inserted in the ‘cold’ storage dewar requires a turbo or diffusion type high vacuum pump; pumping on the insulating vacuum space while the insert is ‘cold’ is not required.

Evacuate the sample tube. Back fill the sample tube with helium gas. It is important to remove the air from the sample tube.

1. Pump the sample tube.
2. Close the vacuum valve at the sample tube outlet.
3. Attach a helium gas supply to the sample tube and back fill.

It is recommended that the gas pressure (1-4 psig) be maintained on the sample tube during the insertion and cooldown. If the pressurization gas line needs to be removed, isolate the sample tube with a vacuum valve mounted on the NW25 insert outlet. Set up the insert for slow insertion and repressurize with the gas pressure hose to the sample tub. This way the two stage regulator will maintain the sample tube pressure during insertion

Helium gas can be supplied using a helium gas cylinder. High purity (‘Medical grade’) 'Welding grade' 99.99% purity helium gas is recommended. To control the gas flow, a two-stage regulator should be used, with a sensitivity of 0-5 psig.

Alternate procedure: When the insert is set on top of the storage dewar and the impedance liquid inlet is immersed in the helium gas space of the storage dewar, the sample tube can then be attached to the mechanical pump. This procedure can be used in place of maintaining a positive (> 1 atm) pressure of exchange gas in the sample tube.

INSTALL INSERT INTO LHe DEWAR

Check to assure that the sample tube is pressurized (1-2 psig) with helium gas. It is recommended to maintain the positive pressure in the sample tube during cooldown by leaving it attached to the two stage regulator. (*Or, follow the alternate procedure noted in the last section.*) Use a flexible plastic hose from the insert to the regulator. The gas pressure is maintained in the sample tube until pumping on the sample tube commences; this will prevent plugging of the impedance.

> Reminder: It is important to replace any air in the sample tube with helium gas before inserting into the ‘cold’ storage dewar.
> Never cool the insert with air in the sample tube.

Before inserting the insert, depressurize slowly the dewar. Avoid contact with any venting cold gas. Remove the storage dewar top quick connect flange. Setup a mechanical support assist; so that the rate of insertion can be controlled. **Slowly,** insert the cryostat directly into the storage dewar. Allow the exiting gas to cool the cryostat and the magnet; Go slow and pause to observe the boil-off gas exiting the storage dewar. The gas amount should be calm and small. When an increase in evolved gas is observed, stop inserting the insert and hold the height until the boil-off settles down. If the magnet is attached to the insert,

remember the magnet represents a large mass that needs to be cooled. Plunging the magnet quickly into the storage dewar will cause excess liquid helium consumption.
Slowly, continue until the insert can be clamped to the top of the storage dewar.

If using a vapor shielded dewar, it is recommended to insert the magnet insert into the dewar before precooling and during precool. Transfer LHe with the magnet insert in place. Then insert the sample insert by itself afterwards.

The sample will cool very slowly, as desired. At this point, the externally supplied helium gas is maintaining the sample tube at positive pressure.

After insertion, vent the dewar's helium gas through the pressure relief installed on the high current leads. Do not allow the dewar to remain directly open to the air; prevent air (ice) from cryopumping into the dewar.

The storage dewar should be vented through the pressure relief at the top of the dewar (~1 psig). Thus, a positive pressure is maintained in the dewar, preventing contaminants from entering; and, the exiting vapor is used to cool the powered leads.

.

WARNING – When handling the insert - be careful - avoid damaging the capillary tube sticking out the bottom. Wear appropriate gloves to protect against the venting gas and cold parts of the insert.

COOL DOWN

With the helium gas in the sample tube and a good insulating vacuum, the sample will cool very **slowly**, as required. Monitor the sample temperature using the temperature controller or readout.

The installed temperature sensors are silicon diode (CD-12A) and RuO2. A calibration table is supplied. Note that most cryogenic temperature controller recommend entering temperature versus log ohms for RuO2 resistance sensors (NOT versus ohms) into the calibration table in the controller.

Cooldown time
& Exchange gas mode of Operation

The cooldown time is slow, by design of the impedance. For faster cooldown, the cryostat can be temporarily operated as a classic two-wall static exchange gas system. Admit a very small controlled partial pressure of helium gas into the insulating vacuum of the insert. A typical 'small' amount is the amount of helium gas volume held by 0.375 in diameter tube x 1 inch long. The exchange gas becomes an adjustable thermal link to the liquid helium bath.

It is important to note that the exchange gas added to the insulating vacuum space may be difficult to remove – a high vacuum type pump is required, i.e., turbo or diffusion.

The exchange gas must be completely removed from the insulating vacuum space to obtain the lowest terminal temperatures.

The exchange gas will be 'cold' and dense. A high vacuum style pump (turbo or cold-trapped diffusion) is required to remove the exchange gas. A mechanical pump cannot be used to remove the 'cold' exchange gas. Do not add exchange gas to the insulating vacuum space of the insert unless a high vacuum pump is available; otherwise, the insert will have to be removed and warmed up to remove the gas.

The impedance has been optimized for low terminal temperature and may be closed at higher temperatures. Sample and insert may need to be cooled to less than 100K before the impedance will open.

When the sample has cooled sufficiently, pump on the sample tube. Liquid helium will be drawn from the main reservoir into the sample region.

Low Temperature Operation

Pump on the sample tube outlet. The fixed impedance has been installed into the bottom of the sample tube. Pumping on the sample tube will draw liquid helium across this impedance into the sample tube. The pump will reduce the vapor pressure of this liquid and hence the boiling point. The temperature of the sample can be lowered to ~ 1.2K, depends on pump used and pumping line lengths and diameter.

Simple to Operate

Continue to pump on the sample tube. The sample will cool to an equilibrium terminal temperature of ~ 1.2K. After reaching the terminal temperature, there will be a short duration where some small oscillatory temperature change may be observed. After which, the temperature will stabilize and maintain for long periods of time - without any operator adjustments needed.

Note: Make sure the pumping line diameter is sufficient large to avoid it restricting the pumping speed (and hence the lowest obtainable temperature). Make sure your pump arrangement or valving will allow the sample tube to be valved off and back filled (pressurized) with helium gas.

STAND-BY

For long unattended operation, sample tube pumping can be maintained and hence the sample just sits at the terminal temperature. However, be aware that a potential problem may occur in the advent of a power failure. When the pump stops, air may be drawn into the sample tube. This may cause some icing problems in the sample tube and might contaminate the sample. The insert will need to be warmed and any moisture removed. If moisture in the sample tube is visible, remove with a 'cloth on a stick'. Remove non-visible moisture by pumping

on the sample tube at room temperature in order to sublime away the moisture. Alternately, the sample tube can be back filled with helium gas. Close the sample tube outlet valve. Attach your helium gas hose and back fill. A pressure regulator is installed on the sample tube to allow closing the sample tube valve.

Be aware that as the back filled gas cools, the pressure in the sample tube will reduce, which is not a problem as long as there are no leaks into the sample tube. Wait and add more gas later to compensate.; or, use the two-stage regulator to hold the sample tube pressure to just above atmospheric. To restart operation, pump on the sample tube.

SHUT DOWN

(i). Turn off all power to any heaters.

(ii). Slowly vent the storage dewar to reduce the pressure to one atmosphere.

(iii). Backfill the sample tube with helium gas.

(iv). Remove the insert from the storage dewar by lifting straight out.

(v). When handling the insert, be careful not to damage or bend the capillary tube sticking out the bottom.

(vi). Make sure that the storage dewar vents through the lowest pressure positive relief.

(vii). Allow the insert to naturally return to room temperature.

GENERAL NOTES
AND SAFETY PRECAUTIONS

Make sure that all sealed and pumpable spaces are protected with safety pressure reliefs.

The inserts insulating vacuum space is protected from over pressure by a safety pressure relief installed at the factory. This relief has been preset at the factory.
Do not remove pressure reliefs or adjust. Never set to a higher pressure.

The inserts sample space is also protected by a safety pressure relief installed at the factory. Do not remove or adjust.

Avoid contact with the cold gases. Note that removal of the positive pressure above liquid helium will result in a quick evolution of COLD gas.

If possible, do not use the same pump on the helium in the sample tube as is used on the insulating vacuum of the insert. Large amounts of helium may temporarily contaminate the roughing pump causing helium to backstream into the insulating vacuum space of the insert.

Back fill the sample tube with helium gas before cooling.
Never allow air into a 'cold' sample tube.

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,
Fax: (603) 621-9960,
e-mail: cryo@cryoindustries.com.