Operating Instructions for the $^3\text{He}$ Insert

William Neils

May 15, 2002

The following are a short list of instructions for running the Heliox $^3\text{He}$ insert in the screen room located in the north east corner of B9. They assume the use of the ITC-503 temperature controller, the left hand side pumping network (with missing regulator) in the screen room, the blue superinsulated dewar on the L-bracket cart with $\mu$-metal shield, and the pumps in the center of B9. These instructions may be modified appropriately to run in other locations with comparable facilities. For additional information and technical drawings refer to the manuals for the Heliox insert and the ITC-503 temperature controller from Oxford.

1 Insert Preparation and Loading

The Heliox insert has 22 leads pre-wired that terminate at the blue connector located on the 1K pot. These wires can be used for sample leads, to run small magnetic field coils, or other devices on a sample stage that do not require large currents. There is a breakout box with six 32 gauge copper leads that can be installed in the auxiliary port if high current leads are required. These six leads should be anchored to the top of inner vacuum can (IVC) and terminate at the home-made potting pins on the 1K pot to reduce heat leaks down the leads. There is also a coax cable that can be installed in the auxiliary port for high-frequency measurements.

The copper sample stage is attached to the $^3\text{He}$ pot with three M3 screws. The leads are plugged into the blue plug on the 1K pot. A sample can be mounted on the sample stage using vacuum grease or silver paste. If the sample needs to be isolate from the grounded sample stage, a small square of lab-tissue can be cut and held in place between sample and stage with vacuum
grease. This sample stage has a Helmholtz coil that can be installed. If high fields are required, the Helmholtz leads should be connected to the auxiliary copper leads through the pins on the 1K pot. All leads should be tied down with thread or dental floss to prevent a thermal touch to the vacuum can.

Prior to closing the IVC the lead resistances and resistance to ground should be checked. Lead resistances should be below 8 kΩ.

1.1 IVC Pump-Down

Attach a turbo pump station to the IVC port at the top of the 3He insert. Grease the cone seal at the top of the IVC on the insert. Slide the can onto the insert and seat the cone seal. Evacuate the IVC and pump for at least 45 minutes. This removes any residual gas from the charcoal sorb located on the 1K pot. If it is detrimental to the sample to be pumped at room temperature, the sorb can be removed and pumped separately. The insert can now be loaded into the room temperature dewar.

2 Initial Cool Down

Prior to cool-down the dewar and insert should be wheeled into the screen room. Attach a pumping line to the 1K pot pumping port and attach the helium recovery to the helium exhaust port. If you intend to remove the insert while the dewar is still cold, you should attach a valve to the 1K pot pumping port. Also, attach the cables from the ITC-503 and the lead cable to the top of the insert. You can now monitor the sample temperature using Sensor 3. Attach a DMM to the gray cable at the back of the ITC-503 to read the resistance of the 1K pot thermometer. A small amount of 4He should be leaked into the IVC to speed up the cooling process. DO NOT USE THE FULL AMOUNT OF HELIUM SPECIFIED IN THE MANUAL. Use approximately the amount of helium in a 6 inch section of hose at atmospheric pressure.

Pre-cooled the insert and dewar with liquid nitrogen. This is added at the top plate through the helium transfer port. The sample temperature should reach 77K in about an hour. Once this temperature is achieved, remove the nitrogen by sliding a long tube through the helium transfer port that reaches the bottom of the dewar. Pressurize the dewar with gaseous nitrogen and blow the liquid into another dewar. Once the liquid stops flowing, reverse
the flow and blow gaseous nitrogen down the tube to the bottom. This warm nitrogen with boil off any remaining liquid.

The dewar should now be hooked up to the helium recovery line. Liquid helium can now be transferred into the dewar. The sample temperature should reach 5K prior to the dewar being filled. Once the dewar is filled and the valves have thawed, close off the recovery line from the dewar and make sure the helium exhaust port at the top of the insert is open to the helium recovery line. The insert is now ready for operation.

3 Insert Operation

This insert works best when holding different temperatures. It is difficult to sweep the temperature of the insert over large temperature ranges. If this is required, it is best to cool the insert to the desired base temperature and add heat to raise the temperature at the desired rate. If the insert is run as outlined below, I have found that the insert has an operational range from 0.35K to 90K without needing to raise the insert out of the helium bath as the manual suggests.

3.1 Low-Temperature Operation

Low temperature are reached by first liquifying the $^3\text{He}$ into the $^3\text{He}$ pot and then pumping on the liquid using the $^3\text{He}$ sorb. With the sample at 4.2K open the needle valve to the 1K pot approximately $\frac{1}{4}$ turn. This should reduce the temperature of the pot to 1K.\footnote{The 1K pot temperature can be monitored by reading the resistance of the uncalibrated 1K pot thermometer. At 4.2K the resistor reads approximately 2.8kΩ while at 1K, the resistor reads approximately 4kΩ.} Next, heat the $^3\text{He}$ sorb to release the $^3\text{He}$. On the ITC-503 change the Heater and temperature Sensor both to 1. Add a small amount of heat (between 4 and 6) to raise the temperature of the $^3\text{He}$ sorb to approximately 45K. The $^3\text{He}$ will begin to condense and the sample temperature should drop to 1.8K. At this point you can read the sample temperature using Sensor 2. If you desire to maintain this temperature, simply maintain the $^3\text{He}$ sorb temperature at 45K.

The base temperature of the Heliox insert can now be reached by cooling the $^3\text{He}$ sorb. Turn off the heater and open the sorb needle valve approximately 1 turn. The sorb temperature (Sensor 1) should drop quickly and
with it the sample temperature. The sample temperature should quickly reach 0.35K. Keeping the sorb and the 1K pot cold should maintain this base temperature in excess of 1 hour with a moderate heat load. Be careful to monitor the sorb needle valve as it will freeze. Once the sorb temperature drops below 10K it does not require as much helium to remain cold. You can close the needle valve to $\frac{1}{2}$ turn.

### 3.2 High-Temperature Operation

Temperature above 4.2K are achieved by keeping the IVC sorb and $^3He$ sorb cold while heating the $^3He$ pot. Open both needle valves $\frac{1}{4}$ turn and let them cool. Add heat to the sample (heater 3) and heat to the desired temperature. For temperature above 20K it will be necessary to open the 1K pot needle valve to $\frac{1}{2}$ a turn.

### 3.3 Thermal Cycling

It is often necessary to raise the sample temperature above $T_c$ to remove trapped flux. In this case you should follow the instructions for high-temperature operation to raise the temperature. To cool the insert again you should close both needle valves completely and add a small amount of heat to the 1K pot (about 4.5 to 5.5 into heater 2). This will heat both the IVC sorb and the $^3He$ sorb releasing gas and cooling the sample. While cooling, monitor the $^3He$ sorb temperature (Sensor 1) and do not let the temperature rise above 55K. Adding too much heat will slow down the cooling process. It is a balancing act.

### 4 Notes

Once filled, the dewar will hold helium for 12 to 16 hours. You can run the $^3He$ insert for multiple days in a row by refilling the dewar with liquid helium every morning provided the sample temperature has not risen above 120K or so. This is usually not a problem if you use the liquid $^4He$ sparingly opening the needle valves only the minimum amount.

When you are finished running, wait for the dewar to warm before disconnecting the 1K pot pump line unless you have installed a valve. Then you may remove the insert whenever you wish.
There is a large tube with a Pb shield around it that is placed inside the dewar. This provides magnetic shielding while cold. You should use this tube when using the Helmholtz coil. A shield installed directly over the IVC will squash the field and change the calibration.