

Helium-3 System Operation

Revised 6/11/95 D.Stewart

latest 10/9/97

Table of Contents:

- A. Loading Sample onto Insert
- B. Loading Insert into Dewar
- C. Cooling Dewar and Insert (300K to 77K)
- D. Cooling Dewar and Insert (77K to 4K)
- E. Liquid He Transfer (Dewar at 4K)
- F. Condensing the He3 (4K to 400mK)
- G. Temperature Control (400mK to 100K)
- H. Magnet Operation

A. Loading Sample onto Insert

1. Place He3 insert horizontal on workbench on foam supports.
2. Remove vacuum sealed IVC can.
 - i. Admit air through IVC valve
 - ii. Screw impact hammer (find in He3 drawer) onto bottom of IVC can
 - iii. Gently knock cone seal free; unscrew impact hammer
 - iv. Carefully remove IVC can
3. Load sample into socket. Test electrical connections.
4. Load and seal IVC can.
 - i. Wipe vacuum grease from cone seal, both surfaces. Reapply a small amount of vacuum grease uniformly to the insert cone surface
 - ii. Connect turbo pump to IVC valve
 - iii. Carefully mount IVC can, push to seal and begin roughing
 - iv. Leak check system
5. Secure He draw tubes to IVC exterior with dental tape and metal tape. Also secure IVC can to insert upper with dental tape. A barber pole wrapping works well.

B. Loading Insert into Dewar

For Warm (300K or 77K) Dewar:

1. Orient insert rotationally to align He3 dump centered over red line on dewar top flange. Alternatively, align 1K pot valve centered over the three magnet lead blow off flow meters.
2. Lower insert into dewar. Resistance will be caused by He draw tubes catching on sleeves and guides inside the dewar. Do **not** continue lowering, you will bend the draw tubes. Instead, check rotational alignment, raise slightly and try again.
3. Clamp top flange, connect rotary pump line.

For Cold (4K) Dewar:

1. Pre-cool insert in tall open LN2 dewar.
 - i. Add He to IVC as exchange gas. Cooling rate of insert is nonlinearly proportional to amount of exchange gas added. For slow cooling, admit very little exchange gas (or none, cools to 77K in ~1.5-2 hr). For fast (30 min) cooling, add 1 surgical tubing foot of He.
 - ii. Lower insert into LN2 dewar.
 - iii. Connect device and monitor thermometry; cool to 77K. Note that the Sorb thermometer is not very accurate in this temperature range.
 - iv. At 77K, flow He gas through the 1K pot and sorb pumping lines to make sure that these are not blocked (if either one is, it's much better to warm from 77K than from 4K). You should see bubbles rising in the LN2 when you open either needle valve. Prevent LN2 flow back into these lines by closing the needle valve before removing the He gas (or closing the pot pumping valve). You should leave both lines full of He gas.
 - v. Pump out exchange gas if desired; if it takes some time to go into LHe, this will prevent the sample from warming, but in general, it's more trouble than it's worth.
2. Load insert into He cryostat dewar.

Note: This process should be done quickly to prevent warming and ice formation on the IVC exterior. Wipe ice if possible before and during insertion.

A. Slow Lowering

- i. Slide top flange seal on insert down as far as possible.
- ii. Begin lowering insert into dewar. Respect rotational alignment described above for warm dewar.
- iii. Clamp top flange as soon as possible. Use large stopper clamp (find in He3 drawer) to support insert at this level. Connect rotary pump line.
- iv. Re-admit He exchange gas to the IVC.
- v. Continue lowering insert gradually, monitor temperature and He boil off. This lowering should take approx 1 hour, depending on patience.
- vi. Pump out exchange gas as insert cools below 10K. Monitor both Sorb and HighT resistors.

B. Quick Lowering

- i. Lower insert into dewar respecting rotational alignment described above.
- ii. Be very careful of the pot and sorb pick-up tubes as these will bend easily. If you feel resistance to lowering, stop, pull back the insert and try again, checking that the insert is straight and aligned correctly.
- iii. If there is still exchange gas, connect the pump soon after the insert is in and begin pumping on the IVC at about 10-20K.

C. Cooling Dewar and Insert (300K to 77K)

1. Disconnect He recovery line at joint between aluminium tee and 1lb check valve; cap open tee port with a blank. Also disconnect pumping manifold at joint between aluminium cross and brass globe valve; support the weight with the rope hoist.
2. Fill dewar with nitrogen.
 - i. Use 5 foot stainless fill tube, insert into fill port on dewar top flange.
 - ii. Fill with LN2 until overflow exits through open brass globe valve.
3. Cool to 77K
 - i. Connect and monitor themometry.
 - ii. Cooling rate of insert is nonlinearly proportional to amount of exchange gas added. For slow cooling, admit very little exchange gas. For fast (30 min) cooling, add 1 surgical tubing foot of He.
 - iii. Before removing nitrogen you may want to pump out the IVC exchange gas.
4. Remove nitrogen from dewar
 - i. Use 5 foot stainless fill tube, be sure that tube is almost touching the dewar bottom to ensure maximum LN2 removal
 - ii. Release all gas pressure in the LN2 dewar
 - iii. Reconnect pumping manifold at aluminium cross / brass globe valve joint.
 - iv. Flow He gas from wall cylinder into dewar, overpressure of ~ 3 lbs will push LN2 back into LN2 dewar. This takes ~ 60 mins. Flow He for a short while after LN2 has stopped to ensure complete evaporation.

D. Cooling Dewar and Insert (77K to 4K)

1. Reconnect He recovery line at joint between aluminium tee and 1 lb check valve.
2. Initial transfer preparation
 - i. Attach the 2 foot transfer tube extension (find in He3 drawer). This injects the LHe to the dewar bottom to allow gas cooling of the magnet mass.

- ii. Approx 40-50 litres of LHe must be available for this initial transfer. The transfer will take approx 2 hours.
3. Transfer LHe as described in section E.
4. Monitor cooling
 - i. Monitor the magnet resistance with an ohmmeter across the magnet leads, it will decrease from ~ 21 ohms @ 300K to ~ 16 ohms @ 77K to ~ 0 ohms @ 4K nonlinearly.
 - ii. Monitor the LHe level with the He level meter set on continuous sampling.
 - iii. Monitor insert temperatures with the SorbT and HighT thermometers. Control cooling rate by modifying exchange gas volume.
 - iv. *Important*. Pump out IVC exchange gas when the SorbT and HighT thermometers decrease below 10K, using the Edwards rotary pump. This prevents condensation and the subsequent drastically extended pumping time. Continue pumping the IVC until base pump pressure of < .01 mbar is reached. This will take several hours, or overnight.
5. Insert will continue to cool to 4K. To speed process if desired, pump on 1K pot and open sorb gas flow.

E. Liquid He Transfer (Dewar at 4K)

0. As of 8/2/96, the level meter is slightly messed up. In theory the length should be set at 29.0 inches, but this does not yield good results. If you have the length set at 100.0 cm, then the cm and % scales read the same thing. Right now (8/2) a value of 50.0 is too little to run (level below L-He intake tubes). A value of ~73.0 is getting reasonable full. When the top of dewar gets really snowy it's time to stop transferring!
1. Transfer preparation
 - i. Check He bag level if He is to be recovered. He bag should be below 0.6 on small DC milliampere meter to left of lab door #127. Please check with Osheroff group to coordinate the transfer with any they are planning. An illuminated green light on the bag meter indicates that the bag is being actively pumped down by a compressor. Press the pushbutton to manually turn on this compressor if it is off.
 - ii. Choose correct transfer tube end cap. Cap with conical tip fits He3 fill gasket. Cap with squarer tip does not.
 - iii. Approx 30 litres of LHe must be available to fill from 15 % to 75 % main bath level. Weigh the intended LHe supply dewar and record initial LHe volume. The transfer will take approx 1 hour.
2. He Transfer
 - i. Insert transfer tube into He supply dewar.
 - ii. Allow gas to flow through transfer tube until it is cold and LHe is about to flow.
 - iii. Insert tube into cryostat dewar through fill gasket adjacent to He level meter electronics connection.

- iv. Flow He gas from wall cylinder into LHe supply dewar. An overpressure of ~ 3 lbs gives a reasonable LHe flow rate.
 - v. **Important**. Continuously monitor the He bag volume content on the DC milliamper meter. If the bag increases over 0.9 on the meter scale then slow or stop the transfer temporarily by closing the input He gas flow and maybe releasing the overpressure. Alternatively, open green Nupro valve below 1lb check valve to blow off excess He gas.
 - vi. Monitor LHe level with the He level meter set on continuous sample.
3. Ending He Transfer
 - i. Release He overpressure in LHe supply dewar.
 - ii. Withdraw transfer tube vertically from cryostat dewar and supply dewar. Cap cryostat dewar and close supply dewar valve as soon as possible.
 - iii. Weigh supply dewar to determine transferred LHe volume.

F. Condensing the He3 (4K to 400mK)

1. Condense cycle preparation
 - i. Check that cryostat HighT thermometer is $\leq 4K$.
 - ii. Check that IVC is well pumped, pressure $< .01$ mbar.
 - iii. Open and run the LabView application "ITC 503" on the Dick desktop. Set the P,I,D feedback parameters to 6,0,2. Select the SorbT as the feedback resistor.
 - iv. Plug the 1K pot resistor input into the ITC 503 rear if desired.
2. Condense cycle
 - i. Start pumping on the 1K pot. Admit short bursts of LHe using the 1K pot needle valve; monitor the pressure on the Pirani gauge. It should rise to scale max when admitting LHe, then slowly pump down. Admit another burst when the pressure decreases to ~ 1 mbar.
 - ii. Open the sorb He gas flow to 40 % using the LabView ITC 503 front panel, with the sorb gas flow on manual control. This maintains thermal isolation between the hot sorb and the cold 1K pot.
 - iii. Heat the sorb to 45K by setting the LabView ITC 503 front panel target temperature to 45K, with the heater control on auto.
 - iv. After the 1K pot reaches 1K and the sorb reaches ~ 45K, begin timing for the 15 minutes required to condense all the He3. Monitor the 1K pot pressure and temperature and admit more bursts of LHe as necessary.
3. Ending condense
 - i. Switch the heater control to manual and decrease heater output to 0 % on the LabView ITC 503 front panel. Wait for the SorbT to cool to $< 10K$.
 - ii. Reduce the sorb gas flow as the SorbT falls. The resting gas flow during cryostat operation at 400mK is 0 - 20 % (0% O.K -Andy) depending on the main LHe bath level. Set the gas flow to maintain the SorbT near 4K. Higher flow is required for a lower main bath level.

- iii. Trim the 1K pot needle valve to stabilize the pressure at 2-5 mbar. This control will stabilize in approx 5 mins as the cryostat hits base T ~ 385 mK. If you want to just run at low temperatures you can disconnect the High T resistor and get a slightly lower bas temp

G. Temperature Control (400mK to 100K)

1. Below the 1K pot temperature (~1.6K), you can set the low-T resistor for feedback. The system will cool by using the sorb heater. For this reason you must be pumping on the sorb gas line. The (sorb) gas flow line can be set at 0.0% and sufficient gas will flow to keep the ends of the sorb at about 7K, which is good enough.

Read the theoretical background on P, I, D control in the Oxford manual and understand it. P is a temperature and I and D are times in minutes. For control below the 1K pot temp, I have found that P, I, D = 2.0, 2.0, 0.5 works well. You need the derivative term to prevent overshoot.

2. Above the 1K pot temperature, feedback off of the high T resistor. This will run both the sorb and He-3 pot heaters. The temperature will rise very easily due to the He-3 pot heater. If there is no He-3 gas floating around (sorb cool) then the temperature will not go down very fast. So we don't want to overshoot with the control. Ergo, I recommend a P, I, D of 20.0, 1.0 and 0.5. Ideally, one can connect a separate power supply up to the sorb and keep it hot so that the temperature can move fast in both directions. This means that the sorb gas flow must be kept on, which uses up more liquid He. I have only gone up to 10K.

DO NOT RUN THE SYSTEM UNATTENDED unless you are sure you know what will happen!

H. Magnet Operation

The magnet can be driven by either the (1) the Keithley 238, which can source up to an amp (2) an AO-6 channel (typically channel 2) and a Kepco Bipolar Amplifier to source up to 15 or 20 Amps or (3) the Lakeshore current supply, which can go the full range (quantum Hall measurements).