

Assorted Notes on Running the Dilution Refrigerator

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The following is a set of notes concerning routine operation procedures for the dilution refrigerator and the fridge dewar. While the instructions are no substitute for first-hand experience, they can serve as a set of guidelines and reminder tips. Hopefully, this can be a working document, in continual evolution to reflect changes in the current protocol. Much of the section on cooling the dilution refrigerator from 4.2 K to base temperature is taken from the Oxford manual but with various additions and specifications. In the following, when I write pot line valve, condenser line valve, pumping line valve, or IVC line valve, I am referring to the valve on the dilution unit itself.

CLEAN OUT THE FRIDGE:

- To begin a new run, strip off the old indium seal and recycle the old indium.
- Blow away indium bits with air gun.
- Scotch brite the two surfaces that will make the indium seal.
- Swab off any dust or spots with a Q-tip and methanol.

BUTTON UP THE DILUTION UNIT:

- Place chip carrier in socket.
- Cover socket with copper tape with a small hole poked in it. Make sure to stick the fiber optic underneath the copper tape if the LED will be used.
- Test connections to sample from BNC boxes. Remember to check the connections periodically while cooling down to save effort if the sample becomes disconnected somehow.
- Screw on the inner radiation seal. These screws need to be only finger tight.
- Form a new indium seal on the IVC can with slightly greased indium wire. Do not locate the joint in the seal close to one of the IVC bolts. Tighten the IVC bolts in small, equal increments, proceeding around in a circle. Wait 30 - 45 minutes and tighten again.
- Pump on the IVC using the leak detector for a few hours. Use the leak detector on the 3×10^{-8} or 10×10^{-8} scale to check for leaks, primarily around the IVC indium seal.
- If the dilution unit has been warm for a long time or has been running for a long time, you can also pump on the dilution unit itself by teeing the dilution unit's condenser line valve and pumping line valve into the leak detector.

PUT THE DILUTION UNIT INTO THE MAIN BATH DEWAR:

- Magnet temperature is at room temperature ~ 173 K, to be measured with a multimeter and the magnet temperature attachment cord.
- Start with all valves on the gas handling cabinet and on the dilution unit closed.
- Lower the dilution unit into the fridge dewar.
- Attach the IVC line, pot line, pumping line, and condenser line to the dilution unit, blowing some He gas through these lines before attaching if desired. The topology of the lines should allow enough freedom to later insert the LHe cold trap.
- Hook up the electrics and the RuO₂ cable. These take a few minutes to warm up.
- Open 4a to pump out the pot line to $\sim 10^{-2}$ mbar.
- Open pot line valve to pump out the pot to ~ 0.2 mbar. Close pot line valve and 4a.

PUMP AND FLUSH MAIN BATH:

- Attach the recovery line to the main bath, since the main bath can only be pumped and flushed through this line.
- Open 4a and 1a to pump out contaminants from the main bath, so that $P_2 < \sim 1$ mbar.
- Always make sure 3a is closed when pressurizing main bath.
- Close 4a and flow He gas to raise $G_3 \sim 1000$ mbar.
- Repeat pump and flush with He gas two more times.
- Close 1a, open 4a to pump line briefly. Close 4a.

ADMIT EXCHANGE GAS INTO IVC:

- A little bit of He gas (the exchange gas) admitted into the IVC puts the dilution unit in thermal contact with the main bath, at room temperature, 77 K, or 4.2 K.
- You do not want to admit air into the IVC along with the exchange gas. Open 5a, 2a, and the IVC line valve to pump the IVC and IVC line to $\sim 10^{-2}$ mbar.
- Close 2a, IVC line valve, and 5a.
- Attach He gas tube onto vent 1 on the back of the cabinet (vent 1 valve should be closed).
- Let He gas flow for a few seconds to blow any air out of the rubber tube.
- Pinch off ~ 1 ft. of tube from vent 1. Open vent 1 valve for a few seconds and then close it to allow only the pinched off gas into the cabinet.
- Open and close 2a. Open and close IVC valve to let exchange gas into IVC.
- Pump on Condenser line and Still line (He-3 line)

TRANSFER LN₂ TO THE FRIDGE DEWAR:

- With a positive pressure in main bath ($> \sim 1000$ mbar) and He gas flowing slightly, insert LN₂ transfer stick into the transfer hole on the fridge dewar.
- The transfer stick should screw in all the way so that the bolt is $\sim 2 - 3$ inches above the top of the transfer hole.
- Shut off He gas flow, disconnect recovery line from main bath, and put a cap on the recovery line.
- Make sure 3a is closed so nitrogen cannot enter the recovery system.
- Connect the rubber tube from the liquid valve on the LN₂ dewar to the transfer stick.
- Start the flow of LN₂, slowly at first, and increasing as the tube stiffens.
- The main bath is full when liquid begins to spill out the where the recovery line is usually attached to. This takes \sim one hour.
- When the main bath is full, stop the flow of LN₂.
- The magnet temperature should be ~ 195 μ for 77 K.
- With exchange gas in the IVC, the dilution unit should cool to 77 K in a few hours, which can be verified by comparing the resistor readings to the typical 77 K readings.
- Check sample connections at 77 K.

REMOVAL OF LN₂: (once dilution unit is at 77 K)

- After the main bath and the dilution unit have precooled to 77 K, the LN₂ must be removed. Be prepared to start a LHe transfer soon after removing the LN₂ because the empty main bath and the dilution unit will begin warming within a few hours.

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- The LN₂ can be forced out of the main bath and back into the LN₂ dewar by raising the pressure in the main bath and reducing the pressure in the LN₂ dewar.
 - Connect the rubber tube from the liquid valve on the LN₂ dewar to the LN₂ transfer stick.
 - Lower the pressure in the LN₂ dewar by opening the gas valve on the LN₂ dewar.
 - To raise the pressure in the main bath, connect the recovery line to the main bath and start the flow of He gas.
 - Open the liquid valve on LN₂ dewar a little at first until the tube hardens, and then open it completely.
 - Leave the gas valve on the LN₂ dewar open to continue lowering the pressure in LN₂ dewar.
 - It will take ~ 1 hour to empty the main bath of LN₂. This main bath is empty when the condensation on the transfer tube turns from clear liquid to snowy white. Another way to verify that the main bath is empty is to crack open the connection to the rubber tube on the LN₂ dewar and see if liquid comes out of the small hole in the side. If it's just gas, then you are done.
 - When done, close the liquid valve on the LN₂ dewar, then the gas valve, and stop the flow of He gas.
 - Once the rubber tube is unfrozen, disconnect it from the LN₂ dewar and use it to blow some He gas to the bottom of the main bath for ~ 2 minutes to ensure that no LN₂ remains at the bottom. Have the green valve on the side of the gas control cabinet open during this time to let the gas escape.
 - Remove the LN₂ transfer tube before pumping and flushing the main bath.

PUMP AND FLUSH MAIN BATH:

- Open 4a and 1a to pump out contaminants from the main bath, so that $P_2 < \sim 1$ mbar.
- Close 4a and start He gas flow to raise G_3 near atmospheric pressure ~ 1000 mbar.
- Repeat pump and flush with He once more if desired.
- About 15 minutes of pumping should bring $P_2 \sim 0.1$ mbar.
- Close 1a, open 4a to pump line briefly. Close 4a.
- Vent the main bath to 1000 mbar of He gas.

-- Now that the main bath and the dilution unit have precooled to 77 K and all the nitrogen is removed from the main bath, do an initial transfer of LHe to the main bath to cool it to 4.2 K (the exchange gas in the IVC allows the dilution unit to reach 4.2 K also).

LHe TRANSFER TO MAIN BATH: (not complete instructions, but just some reminders)

Before the transfer:

- Make sure that 3a is open and that the recovery bag is on (green light lit on the wall box).
- Check that the tips on the two ends of the LHe transfer tube are on the correct ends. For an initial transfer, the tip that opens straight down should go into the main bath. For a noninitial transfer, the tip which opens to the sides should go into the main bath. For an initial transfer, there is no liquid in the main bath, and it is best to spray the LHe to the bottom of the main bath. For a noninitial transfer, there is already LHe in the main bath, so it is better to spray the incoming LHe to the sides to avoid excessive splashing.
- Put the level meter sampling rate on high.

During the transfer:

- For a normal transfer, pressurize the LHe dewar with He gas to ~ 3 psi, then stop the gas flow and wait for pressure to drop to ~ 2 psi before starting flow again. These pressures are not crucial.
- For an initial transfer, the main bath is only at 77 K, so any LHe transferred into the main bath will boil off rapidly and fill the bag. Use a very slow flow rate of He gas, just enough to let the magnet temperature cool without filling the bag too fast. Cooling from ~ 195 ° (77 K) to ~ 300 ° can take ~ 1 hour, but cooling the rest of the way to 1054 ° (4.2 K), takes only ~ 10 minutes.

-- While transferring, you can judge whether there is any flow by watching to see if the recovery line frosts up or not.

-- Make sure the bag does not go to more than 0.75 - 0.80 while transferring, or else stop the transfer by releasing the pressure in the LHe dewar, first to the yellow recovery line and then to atmosphere.

-- Watch out for spurious readings by the helium level meter, including any rapid jumps due to splashing. If it does jump suddenly, you can find the true level by shutting off the He gas flow, releasing the pressure from the LHe dewar, and waiting a minute or two to get the true level.

After the transfer:

-- Put the level meter sampling rate back on low, since frequent level readings will increase the boil off rate from the main bath.

-- Thump the LHe dewar to find the remaining liquid level. If less than a few inches, do a transfer into the LHe dewar within a few hours so that it does not warm.

-- Now that the main bath is full of LHe, compare the resistor readings with the typical 4.2 K values.

-- Check sample connections at 4.2 K.

-- Vent the pot to the 4.2 K main bath by opening 1a and the pot line valve.

-- Turn on the film heater to ~ 15 mW to keep the sorb pump virginal until it is needed for removing the exchange gas from the IVC.

-- Connect the leak detector to the metal tubing which is teed into the condenser line and pumping line.

-- Use the leak detector to pump out the condenser line and pumping line through the two valves on this tee as well as 1 and 3. Pump for ~ 1/2 hour so that P₁ is virtually as low as possible. The condenser line valve and pumping line valve should still be closed until all the air from the condenser line and pumping line has been removed, so that it cannot flow into the dilution unit, freeze, and cause a block.

-- Once the pumping line and condenser line are well evacuated, open the pumping line valve and condenser line valve to pump on the dilution unit for 30 minutes to an hour.

-- Check that the sealed rotary pump is on.

-- Close the pumping line valve and condenser line valve and also close 1, 3, and the two valves on the tee to stop pumping. Disconnect the leak detector.

GETTING FROM 4.2 K TO BASE TEMPERATURE:

-- Insert the LHe cold trap.

-- The thermal contact between the dilution unit and the main bath must now be broken or the dilution unit will not be able to cool further than 4.2 K.

-- Open 5a and IVC line valve to pump out exchange gas for ~ 30 - 45 minutes so that P₂ decreases to ~ 6 x 10⁻³ mbar.

-- A cold sorb will pump gas out of the IVC but not a warm sorb. While pumping on the IVC through 5a and the IVC line valve, leave the film heater at ~ 15 mW. The sorb's pumping is reversible (if it is allowed to cool a little, it will suck in some gas, which it expels if warmed).

-- Close IVC line valve and 5a. Turn sorb heater off so sorb finishes pumping on IVC for 40 - 60 minutes.

-- Fill the LN₂ cold trap. During operation, fill it once a day or so.

-- Start with all cabinet valves closed, except 3a. The pot needle valve should also be closed.

-- Open 1.

-- Open 13a and let gas from behind the pump cool in the LN₂ trap for ~ 1 minute.

-- Open the pumping line valve but keep the condenser line valve closed for now.

Condense the ³He/⁴He mixture

Cool the 1 K pot and introduce the mixture:

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- Check that 1a and 5a are closed.
 - Open 4a and pot line valve to pump 1 K pot. Open the needle valve slightly, and set the throughput to allow the pot to cool. The pot resistor should approach 1.58 k Ω with $P_2 \sim 10 - 20$ mbar. The temperature should settle within a few minutes. This should be checked periodically during condensation, and the needle valve setting should be altered to suit.
 - Condense the mixture from the storage dump as follows. Open 3 and 1 to equalize pressure in the still and condenser lines. Open 9 and the dump valves (*carefully*), and the pressure in the dump is then indicated by $G_2 \sim 460 - 480$ mbar. Check that 13a is open.
 - Open valve 12a. The pressure on G_1 should equalize with G_2 around 400 mbar.
 - G_1 and G_2 should slowly fall as the mixture condenses down the pumping line. No mixture will go down the condenser line since the condenser line valve is closed. Wait $\sim 1 - 2$ hours so that $G_1, G_2 \sim < 50$ mbar. This process is expedited if the pot is cool (1.52 - 1.58 k Ω) with $P_2 \sim 10 - 20$ mbar, so keep a watch on the pot resistor value.
 - The still should now start to cool to about 1.2 K (795 μ , though it won't get there yet); check that the value of the resistor is changing, indicating the still is cooling.
 - When G_1 and G_2 are sufficiently low, close 3 and open the condenser line valve.
 - *Note that the condensation procedure so far is somewhat different from that in the Oxford manual. By letting the gas through 12a to condense only in the pumping line, not the condenser line, impurities in the mixture or the lines will freeze out on the walls of the relatively thick pumping line, not the thinner condenser line, so that a block is less likely.*

Start circulation of the mixture:

- Start to pump on the still by opening valve 6 slowly. Make sure that valve 6 is not opened so quickly that pressure G_2 rises too quickly. Keep $G_2 < \sim 200$ mbar. A small amount of the mixture will be returned to the dump vessel during this operation, but it will condense in again over the next few minutes. When 6 is fully open, wait for G_1 and G_2 to drop, as most of the mixture is condensed into the cryostat. When it reaches a steady value ($\sim 25 - 75$ mbar), close 9, and slowly open 10 to pump the mixture out of the dump vessel. Keep P_1 between 1 - 2 mbar while opening 10. When 10 is fully open (could take an hour), and pressure P_1 is below 0.5 mbar, the dump is empty. Valve 10 should now be closed. If you go too fast in opening 10, so that $G_1 > 200$ mbar, you can put some of the mixture back in the dumps if you close 10, open 9, close 9, and start opening 10 again.
- *Leave the valves on the dump open so that, in case of emergency, the mixture can go back into the dumps.*
- By this time, the still should have cooled to a temperature significantly below 1.2 K, and it will be followed by the coil and mixing chamber. When the system has cooled far enough, the phase boundary is set up in the unit, and the dilution process will start. The change in the mode of operation happens smoothly and automatically, and it will not be noticed.
- When the pressure in the still line falls below 0.3 mbar, the circulation rate should be increased by turning the still heater on to ~ 2 mW so that $P_1 \sim 0.1$ mbar.
- Check again that the 1 K pot temperature and pressure are satisfactory.
- Never leave the dilution unit in a configuration with 9 or 10 open overnight.
- While running, keep occasional watch on the pot resistance and pressure P_2 . You may need to nudge open the needle valve ~ 1 time per day to keep the pot from running dry and crashing the dilution unit. If the pot resistance is between 1.54 - 1.57 k Ω and P_2 is glugging around 10 mbar, then the pot should be stable.

WARMING UP THE DILUTION UNIT AFTER FINISHING A RUN:**Return mixture to dumps:**

- Turn on maximum still and mixing chamber heaters to help force the $^3\text{He}/^4\text{He}$ mixture out of the dilution unit and back into the storage dumps.
- Put the dilution unit in single shot mode (interrupt the usual evaporation/condensation cycle of the dilution unit) by closing 10 and 13a and opening 5 and 9 to pump on dilution unit from both sides for 15 - 60 minutes until all the mixture is back into the dump.
- If $G_2 \sim 400 - 480$ mbar and $P_1 < 0.1$ mbar, then the $^3\text{He}/^4\text{He}$ mixture is out of the dilution unit and back in the storage dumps. It can take a few minutes after G_2 reaches its maximum reading before P_1 bottoms out. Note that G_2 can vary with the atmospheric pressure, so that the most important indication of whether all the $^3\text{He}/^4\text{He}$ mixture is out of the dilution unit is that P_1 is low.
- You can do the next three items while you are waiting for all the mixture to return to the dump.
- Close 4a. Let 1000 mbar He gas into pot line from main bath through 1a. Leave 1a open.
- Open 5a, 2a to pump out IVC line to $\sim 10^{-2}$ mbar (the IVC line valve should still be closed). Close 2a and 5a.
- Admit exchange gas into IVC (so that the dilution unit will warm to 4.2 K) and turn on maximum sorb heat.
- Once the mixture is all back in the dump, close 1, 4, 5, 6, 9, 12a, the two dump valves, the pumping line valve, and the condenser line valve.
- The film/still/mixing chamber heaters can be turned off when all the mixture is out of the dilution unit.

Finishing up:

- Pull LHe and LN_2 cold traps out.
- Open 5a, 2a, 7, 2, 12a, 11a, 1 to pump on both cold traps and remove the impurities released while cleaning the cold traps.
- Clean both cold traps by heating them with heat gun until they are warm and no water is visible on them.
- Close 1, 11a, 12a, 2, 7, 2a, 5a.
- Close needle valve and 1a and open 4a to evacuate the pot to ~ 0.2 mbar. Close pot line valve and 4a. There should not be any He left in the pot while the dilution unit warms up.
- Let 1000 mbar into pumping line and condenser line through vent 1, 7, 2, 1, and 3 (make sure 12a stays closed), so that on the next cooldown, these lines will contain mostly He gas rather than air.
- Close vent 1 valve on the back of the gas handling cabinet.
- Close 1 and 3 and pump out G_1 pressure through 5a, 2a, 7, and 2 so there He gas will not be left in the cabinet. Close 2, 7, 2a, and 5a.
- Open 1a to vent pot line to 1000 mbar of He gas. Close 1a.
- Break all 4 lines and put on covers.
- Disconnect the electrics.
- Remove the dilution unit from the main bath.
- Let the dilution unit warm up thoroughly ($\sim 2 - 3$ hours with a heat gun) before removing the IVC to prevent condensation from forming on the parts inside the IVC.