Hitchhiker's Guide to the Dilution Refrigerator

Version 2.00
Revised 21 August 2004

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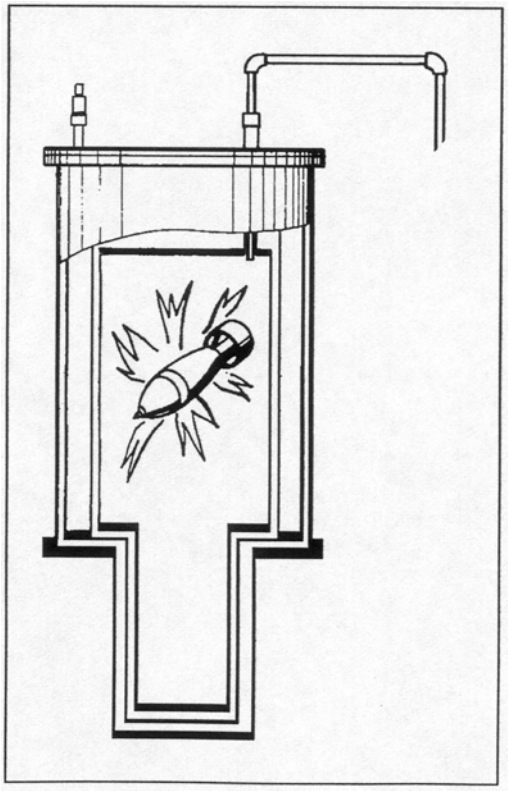

DON'T PANIC

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INTRODUCTION

DON'T PANIC. A dilution refrigerator, at first blush, appears to be a horribly, intricately, awfully twisted labyrinth of tubes and valves and KF flanges designed with the sole purpose of hastening a physics student's descent into madness. This is, to a large extent, quite true. Nonetheless, much of the mystery of dil fridge operation may be dispelled by a bit of hands-on experience and a few helpful pointers. No compilation of notes can supplant the wisdom of physical experience, but hopefully it can help to alleviate the fear and loathing that often accompany a first encounter with a dilution refrigerator. This guide has been created in the hopes of providing a composite resource useful to familiarize a first-time user and remind an experienced hand. It is based upon a variety of sources, from the Oxford manual to past written notes to the ever-changing knowledge of various group members. We hope that it will remain a fluid document, updated and revised to suit the perpetual changes and developments that emerge in the lifetime of a refrigerator. Many of these notes are designed to be as generic as possible, but where relevant have been specialized for the Kelvinox 100 system known as the "Old Stanford Fridge". All Marcus lab dilution refrigerators follow very similar instructions, but a few steps are optimized for the particularities of the Stanford fridge, and various resistance, pressure, and time values vary from fridge to fridge. Much of this information is contained in (confusing and obscure) detail in the Oxford fridge manual; departures from the standard procedure are usually noted when present.

HISTORY AND PRINCIPLE OF OPERATION

The first commercial 3He/4He dilution refrigerator was produced by Oxford in 1967, 16 years after Loudon first proposed the principle of operation. Oxford has held a monopoly ever since, and has developed correspondingly horrendous customer service. Its fridges are sound and reliable, although its service is certainly not. Such is the extent of the relevant history.

When a mixture of the two stable isotopes of helium is cooled below a critical temperature, it separates into two phases. The lighter "concentrated phase" is rich in 3He, and the heavier "dilute phase" is rich in 4He (confusing, no?). The concentration of 3He in each phase is temperature-dependent. Since the enthalpy of the 3He in the two phases is different, the "evaporation" of 3He from the concentrated phase into the dilute phase may provide highly effective cooling. In a gross simplification, the concentrated phase of the mixture is pretty much liquid 3He, and the dilute phase is effectively 3He gas. The 4He composing the bulk of the dilute phase is inert and noninteracting, and may be neglected. The evaporation of 3He from the "liquid" phase to the "gas" phase cools the sample. This process works even at the lowest temperatures because the equilibrium concentration of 3He in the dilute phase is finite even at zero temperature.

When the refrigerator begins operation, the 1K pot is used to condense the 3He/4He mixture in the dilution unit. It does not cool the mixture sufficiently to form the phase boundary, but simply to bring it to 1.2K. Phase separation may be attained only once the temperature falls below the tri-critical point at 0.86K. This cooling is provided by the still; incoming 3He is cooled by the still before it enters the heat exchangers and mixing chamber. Gradually, the rest of the dilution unit cools to the point where phase separation occurs.

It is important that the 3He concentration and volume of the mixture are chosen correctly, so that the phase boundary occurs inside the mixing chamber and the liquid surface lies in the still. If this is not done, the fridge will not cool to base temperature. Thus it is CRITICAL to preserve the balance of the mixture.

During continuous operation, the 3He must be extracted from the dilute phase (to prevent saturation) and resupplied to the concentrated phase. The 3He is pumped away from the liquid surface in the still, where at ~0.6K 3He evaporates preferentially (1000 times faster than 4He). 3He leaving the mixing chamber is used to cool the returning flow of concentrated 3He in a series of heat exchangers. A room temperature vacuum pumping system is used to remove 3He from the still and compress it before passing it through impurity-removing filters and cold traps (one at

77K, the other at 4.2K) and returning it to the cryostat. The inflowing mixture is pre-cooled by the main helium bath and condensed on the 1K pot. A flow impedance (in the form of a capillary tube) is used to maintain a sufficiently high pressure in the 1K pot region for the gas to condense. The experimental apparatus is mounted in the mixing chamber to ensure adequate thermal contact.

The dilution refrigerator has been designed with a two-part external gas handling system. One part (the circulation system) is dedicated to the circulation and handling of the mixture, and the other (the auxiliary system) to auxiliary pumping operations. Both systems are connected to their relevant components on the cryostat and fridge insert by flexible pumping lines.

CIRCULATION SYSTEM (Fig. 10)
The 3He is circulated by a mist-filtered sealed rotary pump. A digital Thermovac gauge is fitted to measure the still pumping line pressure (P1); pump outlet pressure is measured by an analog gauge (G2). The 3He is further purified by passage through an LN2 cold trap before flowing back into the condenser line of the insert. A second analog gauge (G1) indicates the pressure in the condenser line (when valve 1 is open) and the pressure drop across the cold trap. When the system is not in use, the mixture is stored in an external dump vessel. Access to the dump is primarily obtained through valve 9 and the two valves on the dump itself.

AUXILIARY SYSTEM (Fig. 11)
A small, open rotary pump drives the auxiliary gas handling system. This pump vents to air, and so should NEVER be opened to areas containing mixture. Rather, it is used primarily to pump the 1K pot (pulling 4He gas from the main bath) and clean cold traps at room temperature. The auxiliary system is connected to the circulation system through Vent 2 and Vent 1 on the back of the gas handling cabinet.

COOLING DOWN

UNBUTTONING THE DILUTION UNIT

1) Allow inert gas (He or N2; preferably not air) into the IVC can via the IVC valve.
2) Break the old indium seal by unscrewing the IVC bolts and placing bolts in the two dummy holes along the IVC can top perimeter. Slowly screw each, in turn, until the indium seal breaks and the IVC can may be removed.
3) Strip off the old indium seal and recycle the indium.
4) Blow away remaining pieces of indium with an air gun.
5) Gently scotch brite the two surfaces forming the indium seal.
6) Swab off remaining spots of indium or debris with a Q-tip and methanol. Take care to leave no cotton fibers on the sealing surfaces.
7) Remove the inner radiation seal and unscrew the socket cover; gently remove the old sample.

BUTTONING UP THE DILUTION UNIT

1) Test connections from sample to the BNC box; resistances can be checked against those listed in the fridge book. Be certain to check resistance to ground.
2) Place chip carrier in socket. Check to ensure that the pins make good contact with the chip carrier.
3) Screw on the socket cover. Screw on the inner radiation shield. These screws need only be finger tight.
4) Remove the bottom of the inner radiation shield (four screws) and check to make sure that there are no touches between the cold finger and the inner radiation shield. Replace the bottom of the shield.
5) Seal all openings in the inner radiation shield with copper tape. Make certain that there are a few small holes in the tape to allow pumping.
6) Form a new indium seal on the IVC can with lightly greased indium wire. Do not locate the joint in the indium near any of the IVC bolt apertures. slide the IVC can carefully onto the insert, then begin tightening the IVC bolts. Proceed around in a circle, tightening every fourth bolt in small, equal increments (i.e., gently tighten the first bolt, skip the second and third, gently tighten the fourth bolt, skip the fifth and sixth, etc.). In this way, you will tighten evenly around the circle. Tighten until sealed. Wait 30 minutes and attempt to tighten further.
7) Tie the pot intake line tightly to the IVC can with several loops of floss.
8) Pump on the IVC using the leak detector for several hours or overnight. Check for leaks around the indium seal and the IVC valve.
9) If the dilution unit has been warm or running for a long time, you can pump on the condenser line and still line by teeing a turbo to both the condenser line valve and the pumping line valve. Pump as with the IVC.

INSERTING THE DILUTION UNIT INTO THE MAIN BATH DEWAR

1) Magnet temperature at room temperature is ~173 ohms, measured with a Fluke and the magnet temperature leads.
2) Start with all valves on the gas handling cabinet and on the dilution unit closed.
3) Lower the dilution unit into the fridge dewar.
4) Attach the IVC line, pot line, pumping line, and condenser line to the dilution unit, blowing some He gas through the lines first if desired. The topology of the lines should allow enough freedom to later insert the LHe cold trap.
5) Hook up the electrics and the RuO2 cable. Check the room temperature resistance bridge readings.
6) Open 4a to pump out the pot line to 10-2 mbar. Open pot line valve to pump out the pot to 10-1 mbar. Close the pot line valve and 4a.
7) Blow He gas through the pot and pot line via a valved tee attached to the pot line just outside the pot valve. Open the pot valve and needle valve to flow He at 5-10psi throughout the entire process until 4K is attained.
8) Connect positive bias if desired.
[**Variation on insertion for LN2 pre-cooling or cryostat pre-cooling:** Begin blowing He gas

through the pot system via valved tee while insert is suspended above the cryostat. Once the insert is in the cryostat, close the needle valve, pot valve, and the tee valve. Attach the pot line to the tee and evacuate the line via 4a. Close 4a, reopen the tee valve, wait until the pot line has filled with He gas, then open the pot valve and needle valve to continue flowing He gas through the intake line.]

ADMITTING EXCHANGE GAS INTO THE IVC

1) A small amount of exchange gas (in this case, 4He) is necessary to establish thermal contact between the dilution unit and the main bath in order to cool to 4K. You do NOT want ANY air to enter the IVC along with the exchange gas, as water vapor and N2 in the air will freeze out during cooling and pose an unacceptable heat burden. Connect a turbo pump and a valved tee to the IVC inlet valve. Evacuate the line to the IVC using the turbo for ~15 minutes.
2) Attach He gas tube to valved tee in the line. Close off the turbo. Pinch off ~1m of gas tube, then open the valve to allow the ~1m of gas into the line. Close the valve.
3) Rough out the lines in order to clear the just-introduced He. Repeat once or twice more to fully flush and pump the lines.
4) Rough and turbo the line one final time for several minutes. Close off the pump. Pinch off one foot of gas tube, then open the valve to allow the foot of gas into the line. Open the IVC valve fully. Close the IVC valve. A fraction of this foot of gas will have entered the IVC can; this is your exchange gas. Disconnect the turbo from the IVC valve.

TRANSFERRING LN2 TO THE FRIDGE DEWAR

1) Insert LN2 transfer stick into the transfer hole on the fridge dewar. The transfer stick should screw in entirely at the bottom so that the bolt is ~2 inches above the top of the transfer hole. Tug on the stick to ensure a tight seal.
2) Connect the rubber tube from the liquid valve on the LN2 dewar to the transfer stick.
3) Start the flow of LN2--slowly at first, then open to full as the tube hardens.
4) The main bath is full when the magnet temperature reaches and sustains ~200 ohms for 77K. This should take one to two hours.
5) The dilution unit should cool within one hour of filling, as indicated by resistance bridge readings.
6) Positive bias voltage reading should diminish sharply near 77K.
7) Leave the transfer tube and stick connected in anticipation of...

REMOVING LN2

1) After the insert and main bath have cooled to 77K, the LN2 must be removed. Be prepared to start LHe transfer soon after LN2 removal, as the dewar will warm quickly once empty.
2) LN2 may be forced from 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.
3) Lower the pressure in the LN2 dewar by opening the vent valve on the LN2 dewar.
4) Raise the pressure in the main bath by connecting an N2 gas line to the main vent of the refrigerator dewar (you can use He gas if N2 is unavailable, but N2 is preferable).
5) Open the liquid valve on the LN2 dewar so that liquid may flow out of the main bath, through the transfer tube, and back into the LN2 dewar.
6) It will take ~1 hour to empty the main bath of LN2. When the bath is empty, condensed ice on the transfer tube will melt. When this occurs, close the vent and gas valves on the LN2 dewar and (carefully) remove the rubber transfer tube from the top of the transfer stick (leave the stick in). Cold N2 gas should be flowing out of the top of the transfer tube. Allow gas to continue flowing until the magnet resistance drops to ~198 ohms; in this way you may be certain that all LN2 has been evacuated from the dewar.
7) Remove the LN2 transfer tube.

LHe TRANSFER TO MAIN BATH (Not complete instructions; just a few reminders pertinent to the initial transfer)
1) Check that the outlet end of the LHe transfer tube has the correct nozzle. For the initial transfer, you should use a tip that opens straight down, as it is best to spray LHe to the bottom of the empty main bath during the first transfer. For later transfers use the nozzle with side openings, since it's better to spray incoming LHe to the sides of the partially full bath to avoid excess splashing.
2) Put the level meter sampling rate to high.
3) Transfer philosophies vary, but during the initial transfer it is important to cool the insert as adiabatically as possible to avoid excess LHe blowoff. Use a very slow rate of flow; begin the transfer without pressurizing the dewar, and allow dewar pressure to govern the transfer until the dewars equilibrate. Then pressurize the LHe dewar at a VERY low pressure, ~1-2 psi. Cooling the magnet from 200 ohms (77K) to 300 ohms should take roughly one hour, but cooling the remainder of the way to ~1084 ohms (4.2K) takes roughly fifteen minutes.
4) Usually no more than 11 inches (~50L) of LHe are required to cool and fill the fridge dewar from 77K.
5) Once the transfer is complete, turn the level meter sampling rate back to low to reduce boil-off from the main bath.
6) Compare resistor readings with typical 4K values. Check sample connections.
7) You can begin this step when you begin the initial Lhe transfer. Connect a turbo pump or leak detector to the valved tee into the condenser line and pumping line. Use the pump to evacuate the condenser line and pumping line through the two valves on the tee as well as 1 and 3. Don't forget to open the stupid fat valve (old Stanford fridge only) in the pumping line where it runs past the dumps. Pump for 30 minutes to an hour so that P1 is as low as possible. The condenser line valve and pumping line valve should remain closed until the lines are cleared, as otherwise air can flow into the dilution unit, freeze, and cause an obnoxious block.
8) Once the pumping line and condenser line have been evacuated, open the pumping line valve and the condenser line valve to pump on the dilution unit for 30 minutes to an hour.
9) Close the pumping line valve and condenser line valve and also 1,3, and the valves on the tee. You can leave the stupid fat valve (old Stanford fridge only) open for the rest of the run. Disconnect the pump.
10) Stop flowing He gas through the pot line by closing first the needle valve, then the pot line valve, then the tee valve. Open 4a to pump the pot line down to ~10-2mbar. Open the pot valve to pump the pot down to ~10-1mbar. Close the pot valve and 4a.

PREPARING TO CONDENSE

1) The thermal contact between the dilution unit and main bath must be broken or the dilution unit will not cool below 4.2K.
2) Connect a turbo to the IVC line valve. Pump out the lines to ~10-4 mbar, then open the IVC valve to pump out exchange gas for 3-4 hours or overnight so that the pump pressure decreases to 10-5 mbar. Close IVC valve.
3) Insert the LHe cold trap.
4) Insert and fill the LN2 cold trap. During operation, fill it once a day or so, and take care that the level never falls far below ~50%
5) Start with all cabinet valves closed.
6) Open 1.
7) Open 13a and let gas from behind the pump cool in the LN2 trap for ~ 1 minute.
8) Open the pumping line valve but keep the condenser line valve closed for now.

CONDENSING THE MIXTURE
1) Check that 1a and 5a are closed.
2) Prepare to pump on the pot. Open 4a to pump the pot line down to ~10-2mbar. Open the pot valve to pump the pot down to ~10-1mbar.
3) Begin to cool the pot. Open the needle valve slightly, and set the throughput to allow the pot to cool. The pot resistor should approach 1.62 kOhm with P2 ~ 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.
4) Condense the mixture from the storage dump as follows. Open 3 (and 1, which is already open) 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 G2 ~ 450 mbar. Check that 13a is open.
5) Open valve 12a. The pressure on G1 should equalize with G2 around 450 mbar.
6) G1 and G2 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 ~ 1 - 2 hours so that G1, G2 ~ < 50 mbar. This process is expedited tremendously if the pot is cool (~1.55-1.62 kOhm) with P2 ~ 10 - 20 mbar, so keep a watch on the pot resistor value.
7) The still should now start to cool to about 1.2K; check that the value of the resistor is changing, indicating the still is cooling.
8) When G1 and G2 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.

CIRCULATING THE MIXTURE
1) Start to pump on the still by opening valve 6 slowly. Make sure that valve 6 is not opened so quickly that pressure G2 rises too quickly. Keep G2 < ~ 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 G1 and G2 to drop, as most of the mixture is condensed into the cryostat. In many fridges, at this point it is customary to pump the remaining mixture out of the dump vessel by closing 9 and opening 10. However, the composition of the mixture in the old Stanford fridge is such that the fridge reaches a lower and steadier base temperature if the dump vessel is not emptied entirely. Leave 9 open, and don't touch 10. Under no circumstances should 10 be left open for long periods.
2) Leave the valves on the dump open so that, in case of emergency, the mixture can go back into the dumps.
3) 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.
4) Check again that the 1 K pot temperature and pressure are satisfactory..
5) While running, keep occasional watch on the pot resistance and pressure P2. 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 1.58 - 1.62 kOhm and P2 is glugging around 10-20 mbar, then the pot should be stable. Also check G1, G2 periodically while running. If either rises much above its steady value (~25-75 mbar), it is likely that one of the lines has blocked or the condense has been lost.

WARMING UP

RETURNING MIXTURE TO THE DUMPS
1) Plug in the heater BNCs from the resistance bridge to the insert box. Turn on maximum still and mixing chamber heaters to help force the 3He/4He mixture out of the dilution unit and back into the storage dumps.
2) Put the dilution unit in single shot mode (interrupt the usual evaporation/condensation cycle of the dilution unit) by closing 13a and opening 5 to pump on dilution unit from both sides for 15 - 60 minutes until all the mixture is back into the dump.
3) If G2 ~ 500 mbar and P1 < 0.1 mbar, then the 3He/4He mixture is out of the dilution unit and back in the storage dumps. It can take a few minutes after G2 reaches its maximum reading before P1 bottoms out. Note that G2 can vary with the atmospheric pressure, so that the most

important indication of whether all the 3He/4He mixture is out of the dilution unit is that P1 is low (~10-2 mbar).
4) You can do the next three items while you are waiting for all the mixture to return to the dump.
5) Close 4a. Let 1000 mbar He gas into pot line from main bath through 1a. Leave 1a open.
6) Use the turbo to pump out IVC line to ~ 10-2 mbar (the IVC line valve should still be closed).
7) Admit exchange gas into IVC (so that the dilution unit will warm to 4.2 K).
8) Once the mixture is all back in the dump, close 1, 5, 6, 9, 12a, the two dump valves, the pumping line valve, and the condenser line valve.
9) The still/mixing chamber heaters can be turned off when all the mixture is out of the dilution unit.

FINISHING UP
1) Pull LHe and LN2 cold traps out.
2) Open 5a, 2a, 7, 2, 12a, 11a, 1 to pump on both cold traps and remove the impurities released while cleaning the cold traps.
3) Clean both cold traps by heating them with heat gun until they are warm and no water is visible on them.
4) Close 1, 11a, 12a, 2, 7, 2a, 5a.
5) 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.
6) Close the stupid fat valve (old Stanford fridge only). Let 1000 mbar into pumping line and condenser line through the tee at the back of the shielded room, so that on the next cooldown, these lines will contain mostly He gas rather than air. Make sure you close the valves at the tee when you're done!
7) Close vent 1 valve on the back of the gas handling cabinet.
9) Open 1a to vent pot line to 1000 mbar of He gas. Close 1a.
10) Break all 4 lines and put on covers.
11) Disconnect the electrics.
12) Remove the dilution unit from the main bath.
13) Let the dilution unit warm up thoroughly (~ 2 - 3 hours with a heat gun) before removing the IVC to prevent condensation from forming on the parts inside the IVC.

MISCELLANEOUS OPERATIONAL NOTES

-Nitrogen Precooling: If the cryostat already contains liquid helium and you want to cool the insert from room temperature, it's clearly impossible to precool with LN2 in the cryostat. In order to precool the insert to 77K, leave it on the rack and go through all of the standard preparatory steps, including positive bias and He gas flow. You will need to arrange your setup so that it may be used continuously from the rack to the cryostat, which means running long BNCs, etc., from the shielded room. Place the LN2 precooling can around the insert, so that the bottom of the precooling can is only an inch or so below the end of the insert, and the top of the precooling can is above the top of the IVC. Run the LN2 tube into the precooling can, and fill it with LN2. As soon as you begin filling, cover the top of the precooling can in aluminum foil; this is done in order to minimize ice formation on the insert. Once the precooling can is full, wait an hour or so until the insert has cooled to 77K. Then carefully withdraw the precooling can (this is a two-person job, since the can is now filled with LN2) and quickly transfer the insert into the shielded room and lower it into the cryostat. Lower the insert into the dewar as adiabatically as possible to minimize LHe loss and maximize cooling. Proceed as usual once the insert has cooled to 4K. Nitrogen precooling can be notoriously problematic, so be certain to keep the pot line unblocked throughout the process.
-Vapor Precooling: If the cryostat has recently been emptied of liquid helium but remains cold (between 77K and 4K, as measured by the magnet temperature leads), you can use the cold helium vapor in the cryostat and cryostat's thermal mass to precool the insert. Instead of lowering the insert into the dewar after the initial preparatory steps, leave it suspended on the lines above the cryostat and connect the positive bias and helium flow as usual. Once everything has reached the point where you would normally cool with LN2, lower the insert into the cryostat (again, slowly) and proceed with connecting the lines and electrics. Let the insert cool for one or two hours (until it is at 77K or lower) before transferring helium. You can add exchange gas while suspended or while sitting in the cryostat, but do so at the very start of cooling.

-The two pumping procedures that follow the initial LHe transfer are interchangeable. If you're at the end of the day and want to pump overnight, it's far better to pump on the IVC overnight (totally clearing the IVC of exchange gas) and pump out the pot and condensing lines the next morning than to rigidly follow instructions and do it the other way around.

COMMON PROBLEMS (AND HOW TO FIX THEM)

BUTTONING UP THE DILUTION UNIT

-The indium seal leaks. A large leak will prevent you from adequately pumping down with the leak detector; a small leak will show up in leak detection. Either way, you simply have to break the seal and try again.

INSERTING THE DILUTION UNIT

-Positive bias gives you an enormous leakage current, or one that doesn't diminish upon cooling. This means that your gates are leaky or your sample connections are shorted; check accordingly. It could also be a sign that something's wrong with the switches on your breakout box; check to make sure the switches are still isolated.

REMOVING LN2

-When you try to remove the LN2 from the cryostat, nothing comes out. This could be a sign that the siphon tube has been bent, or that the LN2 transfer tube is not properly seated in the siphon cone.

LHe TRANSFER TO MAIN BATH

-When you transfer, you create a continuous geyser of LHe blowoff and the cryostat refuses to fill...or perhaps, once filled partially, the level drops precipitously back to 0% after the transfer. It is likely that LN2 was left in the bottom of the fridge, either due to problems listed above or inadequate LN2 removal. This constitutes an enormous thermal mass with a large enthalpy of cooling and freezing. Generally, you must let the cryostat warm back to 77K and try removing the LN2.

CONDENSING THE MIXTURE

-You've opened 4a and the pot valve to pump on the pot, and then open the needle valve to set the throughput on the pot...but nothing happens. Your pot intake line is blocked. This shouldn't happen if you've flowed He gas throughout the cooldown, but it has been known to occur. The first thing to do is try a quick-pull operation. Close everything off and break all the lines EXCEPT for the condenser line, as at this point the LHe cold trap is cold and should not be exposed to air. There is enough play in the condenser line to pull the insert without problems. Cap off the pot line after the inlet valve, leaving the inlet valve portion on the insert. Flow He gas into this capped-off volume, opening both the pot and needle valve. You won't get any flow-through; the line's still blocked. Pull the insert quickly out of the cryostat and heat-gun the pot intake line (and ONLY the pot intake line) while overpressuring the He gas to 10-15 psi. The idea is to melt the pot intake blockage and then flow He gas through the line to keep the blockage from re-forming. The line should hiss with He gas outflow once the block has melted. Then, continuing to flow gas, lower the insert back into the cryostat. This should all be done VERY quickly to keep condensation from forming. Once back in the cryostat, reconnect the lines and pump as necessary to clear them, then proceed. Sometimes the block may re-form once you're back into the cryostat. If this is the case, the only way to clear the line is to pull and warm the insert back to room temperature.

-The pot throughput is fine, but the pot won't cool. The easiest time to check this is right when you've started flowing through the pot, before condensing. Condensation puts an extra heat load on the pot, making the pot temperature during condensation an unreliable indicator. Before this, however, you should be able to cool the pot until the pot resistor is > 1.5 kOhm. If the pot won't cool adequately, you will be unable to condense in a finite time interval. There are two common problems that can prevent pot cooling. The first is an inadequately-evacuated IVC can. If some volume of gas remains in the can, the thermal contact with the 4K bath will prevent the pot from reaching 1.2K. This often occurs because the exchange gas has not been entirely removed. Stop pumping on the pot, then pump on the IVC can with a turbo pump for several additional hours or (preferably) overnight. You should never pump on the IVC while pumping on the pot, since the cooling of the pot will make it even harder to remove gas from the IVC. If the pot still won't cool, it's possible that you have a touch from the cold finger to the IVC. Pull the insert, warm up, open the can, and check for touches. Then cool again from the beginning.

-The mixture won't condense; you've waited for several hours and the pressure drops on G1, G2 are only incremental. This often results from an inadequately-cooled pot; check to verify that the pot is cool. It can also mean that you have a touch or an incompletely evacuated IVC, since the mixture won't condense at 4K.

CIRCULATING THE MIXTURE

-While running, G1 and G2 both rise well above 50 mbar. You have lost your condense, which often results from a pot crash. Simply pull the mixture, recondense and recirculate.

-While running, G2 is substantially higher than G1. This likely means that the LN2 cold trap has blocked. Pull the mixture, follow the LN2 trap cleaning instructions, and then recondense and recirculate. Blockages here may indicate a leak in the lines, so you may want to consider pulling and leak-checking the system if the problem persists.

-While running, G1 is substantially higher than G2. This likely means that the LHe cold trap has blocked. Pull the mixture, follow the LN2 trap cleaning instructions, and then recondense and recirculate. This often results from an inadequately-filled LN2 cold trap.

CLEANING THE MIXTURE

(Modified from LDC)

Everything is closed and stable--all mixture is in the dump, all cabinet valves are closed. If the pressure on G2 is higher than its typical equilibrium value (~540 mBar for the old Stanford dilution refrigerator), it is likely that some extraneous gas--probably air--has entered into the mixture. Often times this additional pressure may signal a leak into the back of the pump, but it may also result from single-event process or off-gassing. Regardless of greater problems, it's necessary to clean the mixture of impurities before proceeding. The idea is to trap the impurities by circulating the mixture through the LN2 cold trap via a circuit consisting of the sealed pump, 13a, 12a, and 5. After circulating for one or more hours, you return the mixture to the back of the pump and clean the LN2 trap, which now contains the removed impurities.

Initial configuration: Most of the mixture is in the dumps; dump valves are closed. All cabinet valves are closed.

CIRCULATING THE MIXTURE

1) Fill the LN2 cold trap.
2) Open 13a to allow some of the mixture to enter the cold trap.
3) Open 5.
4) Open 12a very slowly, beginning the circulation at a low rate. Watch the pressure on G1 and ensure that it doesn't rise above ~100 mBar. Also watch the lines leading into the LN2 cold trap. Open 12a until condensation appears on these lines, but no further.
5) Monitoring G1, circulate the mixture for one hour or longer, depending upon the scale of the impurities.

RETURNING THE MIXTURE

1) Close 13a to stop circulation. G1 should drop and G2 should rise accordingly.
2) Open 12a fully, increasing the rate at which mixture is pumped from the LN2 cold trap.
3) Wait ~10 minutes until G1 has bottomed out and G2 is (hopefully) back at equilibrium mixture pressure, ~540 mBar. If it is still above 540 mBar, repeat the entire cleaning procedure several times.
4) Close 12a.

CLEANING THE LN2 COLD TRAP

1) Verify that 12a, 13a, and 4a are closed. Open 5a to pump out the IVC/OVC line.
2) Open 2a, then 7, then 11a. The pot pump is now pumping on the LN2 trap.
3) Remove the cold trap from the trap dewar. Warm the trap and then let it cool to room temperature using a heat gun.
4) Watch P2. It should rise immediately as impurities are released from the trap, then fall back to an equilibrium value.
5) Reinsert the trap to the LN2 dewar.
6) Close 11a, then 7, then 2a, then 5a.

This brings us back to initial conditions, and we may repeat the cleaning process as necessary. If you wish to clean the mixture in the dumps as well as the mixture behind the sealed pump, the following steps must be included in the above directions:

--Between steps 1) and 2) in CIRCULATING THE MIXTURE, open the dump valves and valve 9.
--After step 4) in RETURNING THE MIXTURE, close the dump valves and valve 9.

HELIUM TRANSFER

(Modified from LDC)

BEFORE THE TRANSFER

1) Roll a LHe storage dewar to the doorway of the shielded room.
2) Close the safety venting valve on the LHe dewar.
3) Set the LHe level meter rate to FAST on the Oxford control panel. In this setting, the level meter updates every 30 seconds. This does put a heat load into the system, so make sure you're metering on SLOW at all other times.
4) Remove the blanking cap from the aperture on the overpressure line of the fridge cryostat.
4) At the He inlet flange on the refrigerator cryostat, completely unscrew the black nut/cap that seals the flange. Set the nut down nearby, and take the cap, washer, and o-ring to the LHe transfer tube. Put the cap, washer, and o-ring onto the fridge end of the tube (in the proper order!); it will screw into place when the tube is properly seated during transfer.
5) Make sure that the proper nozzle (the one with side vents) has been screwed onto the fridge end of the transfer tube (for initial transfers, use the direct nozzle as per cooldown instructions)
6) Move the red vacuum tube attachment on the storage dewar end of the transfer tube (the "seal hose") down to the bottom of the tube. Remove the transfer tube from the rack, and carry it over to the shielded room.
7) Hop on a stool. Insert the storage dewar end of the transfer tube into the vertical valve of the storage dewar. This valve should be closed--every valve on the storage dewar should be closed at this point--so you can only insert the tube an inch or two before it hits the valve. Push down the seal hose until it makes a tight seal between the transfer tube and the top of the vertical valve on the storage dewar. This prevents He gas from blowing out of the storage dewar top as you transfer.
8) Start the transfer. Open the vertical valve on the storage dewar and push the storage dewar end of the transfer tube down into the LHe storage dewar. There are a few "sticky" points towards the top of the tube; push and twist to get the tube down. Push until the bottom of the transfer tube hits the bottom of the storage dewar, then pull it up ~1cm or so. You never want the tube to rest on the bottom of the dewar, but you do want to have it close to the bottom. The fridge end of the transfer tube should still be in your hand, with the cap-washer-ring assembly attached. There should be thumping and rushing noises as He gas flows through the tube.
9) Jump off the stool and bring the fridge end of the transfer tube to the He inlet flange on the refrigerator cryostat. Don't put it in yet. If you insert it into the cryostat before LHe begins flowing, you will be blowing hot gas into the bottom of the cold cryostat--not a good idea, unless you like blowoff geysers. After ten to thirty seconds, the nozzle on the transfer tube will begin to frost up, and shortly thereafter LHe will begin to flow out, looking like a white flame. As soon as you see this coming from the tube, insert the transfer tube into the cryostat. Push down quickly until the nozzle hits the siphon cone in the body of the cryostat. If you don't do so quickly, LHe will cause air to freeze in the tube flange and the tube will block. This is BAD (if it happens, you need to stop the LHe flow. Open the side vent on the dewar to depressurize the dewar, remove the transfer tube from the cryostat, and start over after making sure that the tube flange is free of ice). Screw the black cap/washer/ring onto the top of the inlet flange. You are now transferring LHe.

DURING THE TRANSFER

1) Set up the pressurization circuit for He gas. Open the He gas valve on the back wall. Don't touch the regulator unless you want to change the flow rate. Hook up the adaptor to the hose. Connect the adaptor/hose to the side vent on the storage dewar and tighten the bracelet with the yellow hex screwdriver. Leave the screwdriver handy.
2) The initial part of the transfer should take place using only the pressure in the dewar (this is why we kept the dewar pressurized at the beginning of the process). Once the storage dewar pressure drops below 1psi (and the increase on the level meter is less than 0.5% per reading), pressurize the storage dewar by opening the side vent valve and allowing He gas to flow in.
3) Keep the storage dewar pressure steady between 1 and 3 psi. If it rises too quickly, lower the rate on the regulator or close the side vent valve temporarily until the pressure drops again.

4) When the level meter reaches 92%, stop pressurizing the dewar by closing the side vent valve and then turning off the helium gas valve on the wall. The transfer should finish with the pressure remaining in the dewar. Remove the He gas hose.
5) When the level meter reaches 96% (it won't go higher), open the side vent valve to depressurize the storage dewar and stop the transfer.

AFTER THE TRANSFER
1) Put on gloves, unless you're masochistic.
2) Unscrew the black cap/washer/ring assembly from the top of the inlet flange. Don't unscrew it entirely; unscrew it enough so that you can pull the transfer tube out of the cryostat while leaving the cap/washer/ring assembly attached to the inlet flange. Pull out the transfer tube. Put the black nut into the cap/washer/ring on the inlet flange. Pull out the transfer tube from the storage dewar. Close the vertical and side valves on the storage dewar and open the safety valve. MAKE SURE TO OPEN THE SAFETY VALVE.
3) Place the transfer tube on the rack and attach it with clips.
4) Replace the blanking cap on the aperture in the overpressure line of the fridge cryostat. It may let out spurts of He gas, but this should slow and stop in a matter of minutes.
5) Return the helium level meter rate to SLOW.
6) Thump the helium storage dewar and record the new level.

CRYOPUMPING THE MIXTURE

Sometimes, when the fridge is not operating, you may need to open up the circulation lines to check for leaks or modify the system. When not running, the mixture is stored largely in the dump, with some additional volume in the segment of lines between the sealed pump at 13a (read by G2). If you need to open other areas of the lines beyond 13a and before 5, 6, and 10, then it suffices to keep these valves closed and operate on the other lines as necessary. If, however, you need to access the volume between 5/6/10 and 13a (the sealed pump and the line volumes immediately before and behind it), you must first remove and safely store the small volume of mixture behind the sealed pump. This is done with the cryopump, which is usually stored in the pump closet. Follow the cryopumping instructions in the Oxford manual, with appropriate modifications (ask a senior student for help). Once you have removed this volume of mixture, you may open the lines freely. ALWAYS make certain that the dump is closed.

POSITIVE BIAS

The idea with positive bias is to raise the carriers in the 2DEG beneath the gates during cool-down. This leads, as a rule, to quieter dots...although the effects of positive bias remain something of a dark art. Apart from effects during measurement, positive bias allows you to check gate leakage and gate function continuously during cool-down, which is very useful for diagnosing device problems before you reach base temperature. Basically, you simply want to apply a small positive DC voltage to the gates at room temperature. When you start, the insert should be in the cryostat and everything should be at room temperature (if the cryostat is cold and you need to pre-cool or will immediately cool the insert upon lowering it into the cryostat, set up positive bias on the insert at room temperature--even if you have to connect it while the insert is hanging above the cryostat). You want to create a circuit that runs something like [battery box high]-[gates]-[grounded ohmic]-["-" end of splitter]-["+" end of splitter]-[battery box low]. The output of the splitter should be connected to the input of an Ithaco, and the Ithaco output to a DMM. This way, you can apply a constant positive voltage to the gates and read out gate leakage from the ohmics on the DMM. The connection from battery box high to the gates can be done in several ways. The easiest is to connect the battery box high to an unused channel on the breakout box. Flip this channel to bus, then flip all of your gates to bus as well. Remove the grounding caps on the gate channels one by one--this way the bus will remain grounded until the last cap is removed. The bus should now be floating. Turn up the positive voltage slowly, to around 300-400 mV. The Ithaco sensitivity should be 10-7 or 10-8. Record the value on the DMM, and write it down. Figure out the resistance value of gate leakage; it's just V=IR, where V is the input (300-400 mV) and I is the DMM signal, multiplied by the Ithaco sensitivity to yield a current. You now

have positive bias on all of your gates, and may cool down. However, if you want to give a thorough diagnostic, it is best to first test gate leakage for each gate separately. Follow the same procedure as above, but only put one gate to bus at a time. That is, connect the unused channel and put it onto bus. Put ONE gate channel to bus, remove its grounding cap, and turn up the positive bias. Record the room temperature value on the DMM. Turn down the bias again, replace the grounding cap, and take the gate channel off bus. Repeat with all gates. Each gate should have a room-temperature gate leakage on the order of MOhms or higher. If much lower, it's a sign that something's wrong with your gates, your ohmics, your breakout box, or your wiring. Once you've checked each gate individually, follow the above procedure for putting all gates onto positive bias. The total value on the DMM should be slightly lower than the sum of individual gate values. Now you're ready to cool.

Fig. 1: Front side of dilution refrigerator

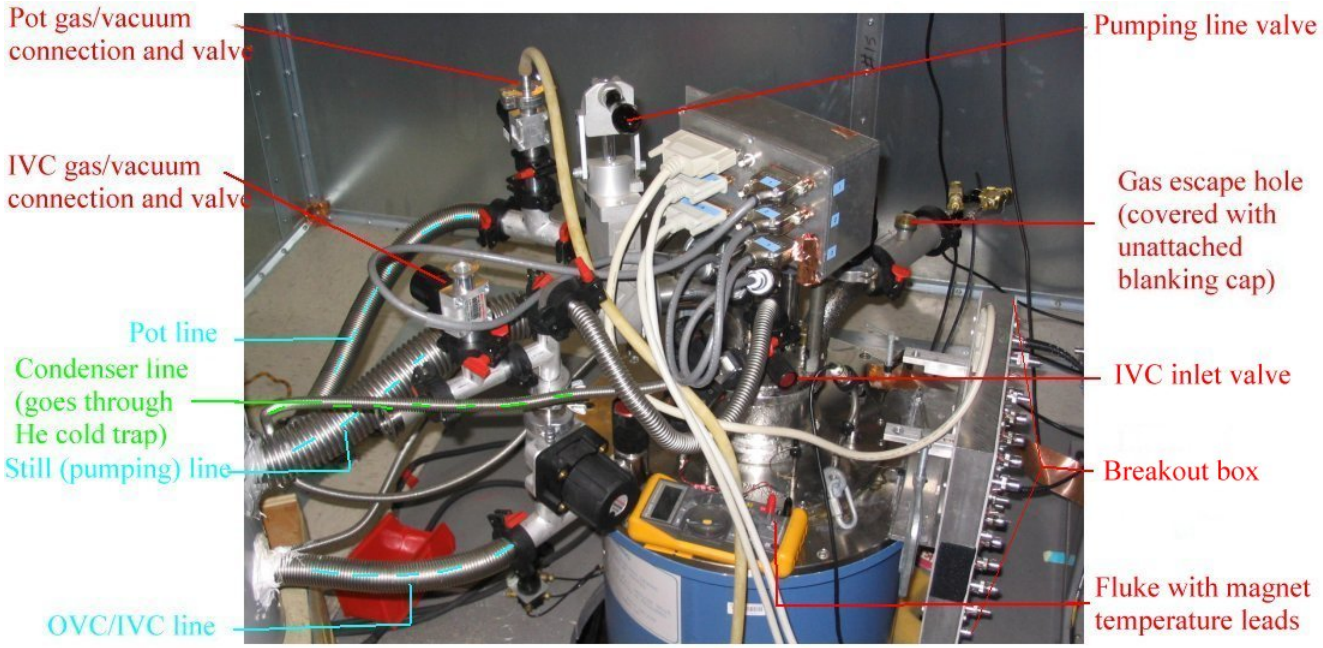

Fig. 2: Back side of dilution refrigerator

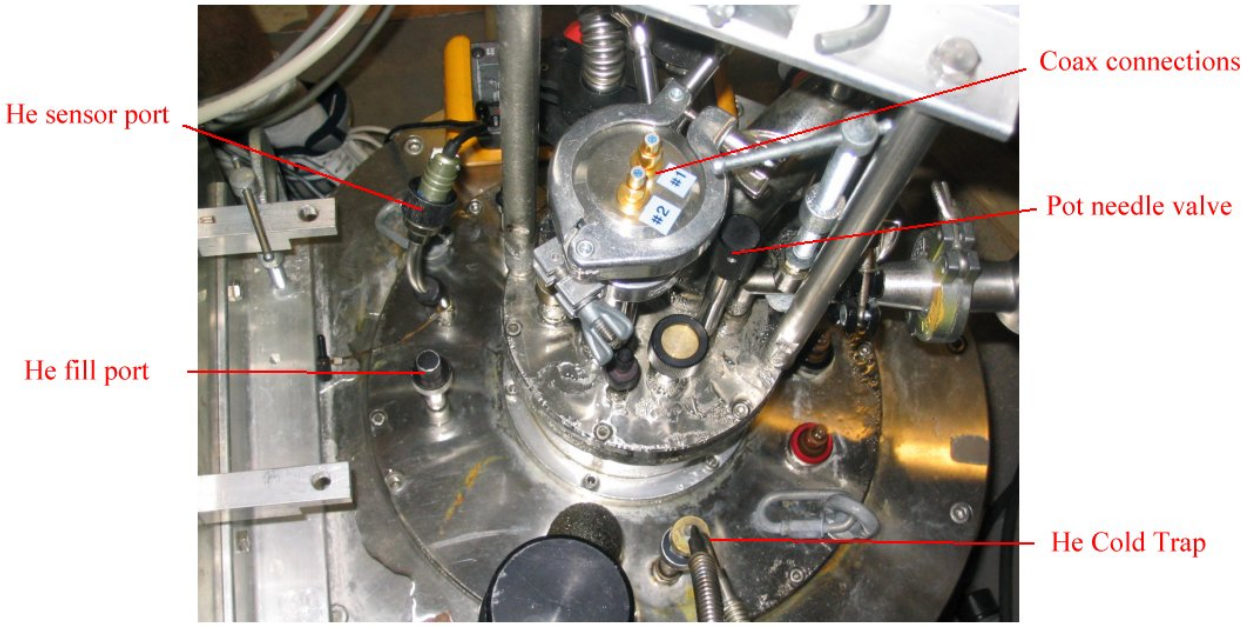

Fig. 3: 3He/4He Mixture Storage Dump

Fig. 4: Outside connections

Fig. 5: Insert without IVC

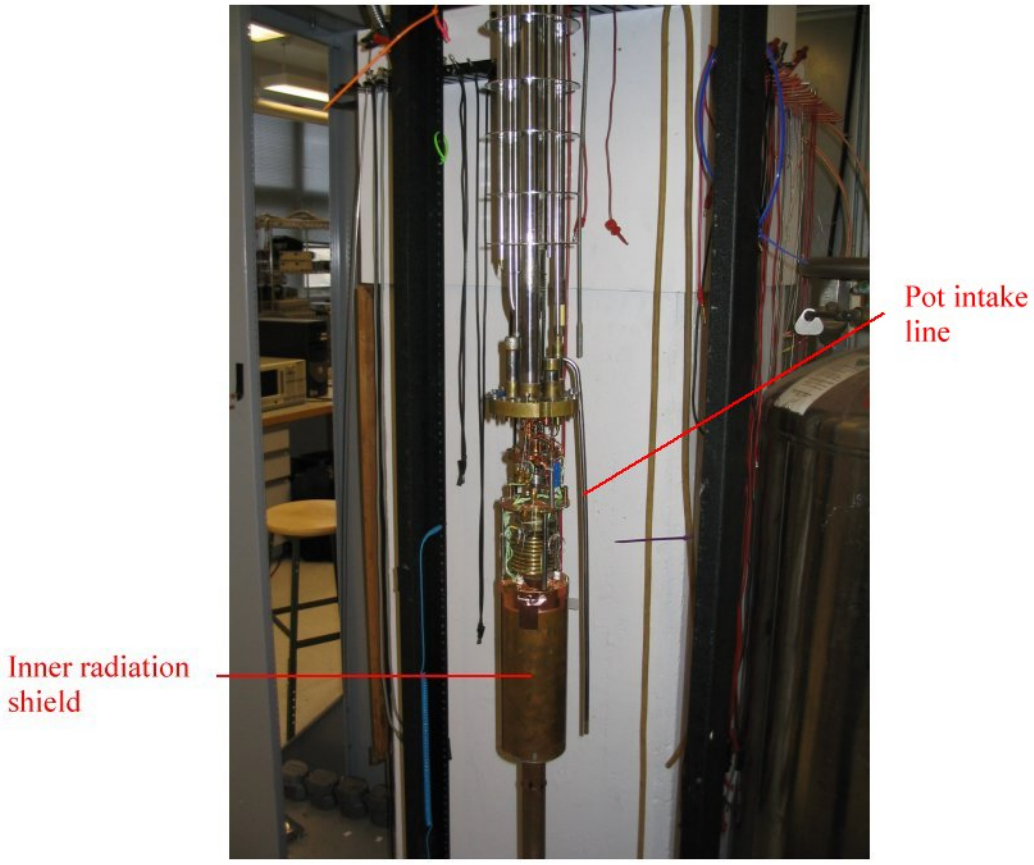

Fig. 6: Indium Seal

Fig. 7: Insert without inner radiation shield

Fig. 8: Flesh may freeze and stick to cold surfaces

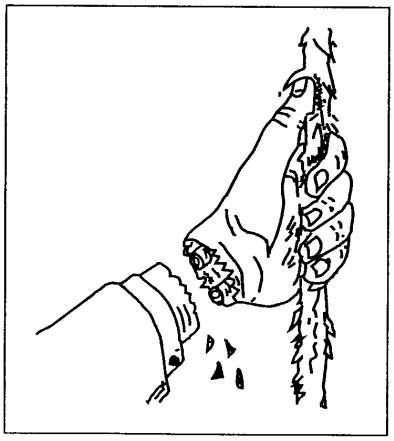

Fig. 9: Schematic diagram of dilution refrigerator

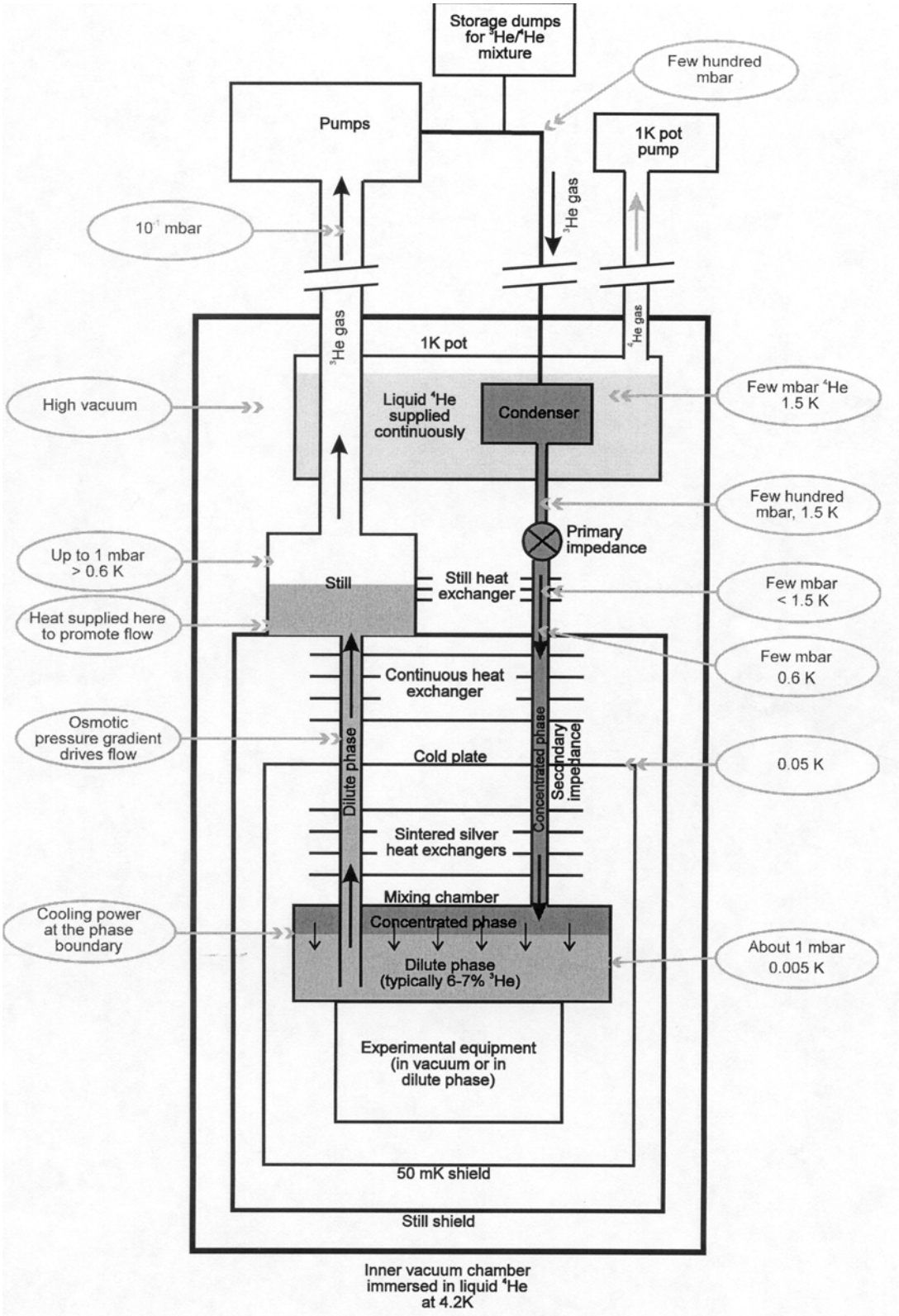

Fig. 10: Circulation system schematic

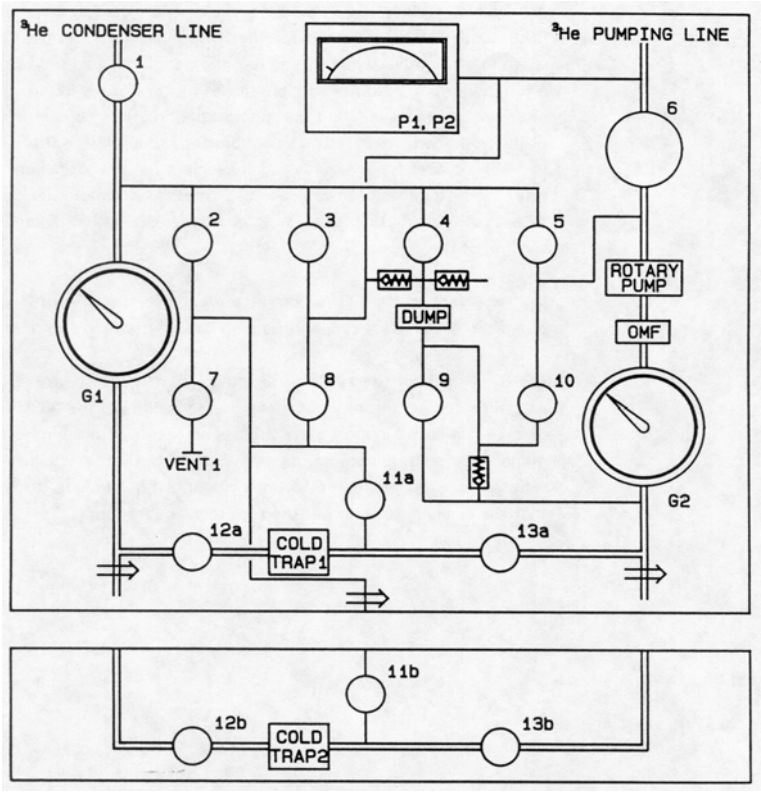

Fig. 11: Auxiliary system schematic