Dilution Refrigerator manual

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Abstract

This document provides a practical guide to dilution refrigerator operation and describes technical details about the hardware. It is intended to help one operate the dilution refrigerator without fatal mistakes.

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Part I

Operation

1. **Terms used in the document**

   IVC Inner Vacuum Can

2. **System preparation prior to cool-down**

   1. Make sure there is no thermal link between each temperature stage. Also any parts in the IVC must not touch the IVC.

   2. Put the IVC on and tighten screws. Make sure an indium wire is right in place during sealing.

   3. (optional)Pressurize 1K pot line with helium gas by around 5 psi and dip the end of 1K tube in IPA to see if bubbles come out.(Verify that 1K impedance line is open.)

   4. Lower the insert down *slowly* into the cryostat. Be careful for the 1K pot tubing not to be bent. Make sure an O-ring is in place.

   5. Connect all necessary lines to the insert, including 1K pot line, condenser, still, cryostat pumping line, thermometer/heater cable, and helium level meter.

   6. Pump He3 gas handling system lines including condenser and still lines, up to low $10^{-5}$ Torr. The cold trap should be still at RT. Once the pumping is finished, put the cold trap in liquid nitrogen dewar.

   7. Start pumping the IVC.

   8. Turn on RGA when the pressure drops low enough (below $2 \times 10^{-4}$) Set it for He3 leak test.

   9. Put some amount of mixture from the tank into still line through cold trap up to $P_1=100$ Torr.

   10. Watch RGA for any He3 leak into IVC from the condenser and still lines.

   11. Do flow measurement(to make sure the dilution fridge is clear of plug): Start pumping only the condensing line. When the condenser line pressure reaches minimum, typically around 100 millitorr, stop pumping and monitor pressure rise.(typically 100 millitorr/min). RECORD the number.

   12. Collect the mixture back into the tank.

   13. Pump the dewar and fill it with helium gas up to 1 atm.

   14. Watch RGA for any He4 leak into IVC from outside IVC.

   15. With the dewar at 1 atm of helium, pump and flush the 1K pot three times with helium gas.
16. Pump and flush the 1K pot three times and then pressurize the 1K pot with helium gas up to about 5 psi.

17. Check the continuity of 1K pot: Stop pressurizing and see if the pressure decreases.

18. Watch RGA for any He4 leak into IVC from 1K pot line.

19. If no problem is found, continue to pump the IVC to low $10^{-5}$ Torr level.

20. Stop pumping and put **Helium4 exchange gas** in the IVC.

3. **Cryostat pre-cooling**

   1. Start transferring L.N2 by pressurizing the L.N2 dewar. Start measuring temperatures at each stage.

   2. Stop when the L.N2 rises up to appropriate level. Monitor the resistance changes in lower and upper resistors.

4. **Check-out at liquid nitrogen temperature**

   1. Check the continuity of 1K pot: Stop pressurizing and see if the pressure decreases.

   2. Pump IVC and turn on RGA when it’s ready.

   3. Introduce the mixture stored in the tank into the still through cold trap. Watch RGA for any leak on the still and condenser line.

   4. Do the same flow measurement as done at RT. Pump the condenser line (100 millitorr) and maintain $P_1=100$ Torr in the still line. Watch for pressure rise when pumping stops, and RECORD the rate.(should be 5 mtorr/sec)

   5. Collect the mixture back into the tank, but leave some amount(several Torr) for thermal contact.

   6. Pressurize the cryostat with N2 gas to back-transfer L.N2 to the liquid nitrogen dewar.

   7. Pump the cryostat and fill it with helium gas. Watch RGA for any leak on vacuum flange.

   8. Check the continuity of 1K pot: With 1 atm helium gas in dewar, pump 1K pot.(minimum may be 150 millitorr.) Stop pumping to see if the pressure rises. The rate should be about 100-200 millitorr/sec. During this step, watch RGA for any change in He4 level due to leak on 1K pot.

   9. Overpressurize the 1K pot up to 5 psi.

10. Do other necessary check-ups.

11. Stop pumping the IVC and put some exchange gas(2 Torr) in.
5. **Liquid helium transfer and check-out**

1. Transfer L.He very slowly initially to make most of cold He gas\(^1\), otherwise the L.He will be wasted a lot.

2. when the L.He starts to collect, keep He pressure in the dewar at \(\sim 3\) psi and keep transferring.

3. Stop transferring and wait till each temperature stage becomes close to 4.2K.

4. **Check if 1K pot is open.** Stop pressurizing and see if the pressure decreases.

5. Pump the IVC to remove He exchange gas.

6. Do bake-out for about 3 hours. Apply the right amount of heat to each temperature stage. Don’t overheat

7. Keep pumping IVC until the helium partial pressure becomes lower than \(10^{-9}\) Torr level.

6. **Condensation and circulating mixture**

At 4.2K, the resistances of each stage are (1K pot, still,H.E, M.C(carbon),M.C(Ge))=(420,1000,150,150,20)

1. Start pumping 1K pot. The resistance is about 600 \(\Omega\).

2. Release mixture slowly from the tank into only still line.

3. Run He3 pump to pump out the mixture from the tank into the still line.

4. Once the tank is empty, shut off the tank and start to pump the still keeping the pressure on the condenser below 760Torr\(^2\). At the beginning of circulation, you will need to open and close the valve between He3 pump and still carefully since the pressure in the still will be high.

5. Turn on the diffusion pump. It will take about 30 minr to have full pumping speed.

6. When the still pressure is below 1000 mTorr, switch to the diffusion pump.

7. Apply heat to the still. About 2 mA to the still will be optimal. At this stage, the TC1, TC2/P1 are about a few hundred millitorr, and less than 100 Torr.

7. **Maintaining the dilution refrigerator during operation**

7.1. **Cold trap**

7.1.1. **Cleaning**

The cold trap can be clogged, so it’s necessary to monitor the status of the cold trap. Check the pressure difference between P1 and P2. At low flow level, if the pressure difference is greater than 50 Torr, the cold trap should be cleaned.

1. Shut off the valves to the condenser and still.

\(^1\)The ice on the He recovery line should extend no more than a few meters.

\(^2\)The hermetic seal on the He3 pump can be damaged if the pressure is over 760 Torr.
2. Collect the mixture residing in the gas lines into the He3 pump. (If the pressure gets high at the exhaust port, dump some mixture into the tank.)

3. Start pumping the cold trap before the cold trap is taken out of L.N2 dewar.

4. While pumping the cold trap with only roughing pump, heat the cold trap with a heat gun. The cold trap is hard-soldered, so using the full power of a heat gun is OK.

5. Watch the pressure rise and once the pressure starts to decress, you may stop heating the cold trap and start running the turbo pump.

7.1.2. refilling L.N2

When the L.N2 level is low in the dewar, transfer L.N2 from another L.N2 dewar to it. Do not take the cold trap out of the dewar.

7.2. Relief valve

It allows us to operate the refrigerator unattended (i.e. overnight). The relief valve opens when the pressure difference across the valve is about 250 Torr. In case the back pressure of the He3 pump becomes high, the mixture is automatically collected in the tank, not being lost. Let the relieve valve open when the system needs to operate unattended.

8. Warm-up

1. Stop 1K pot pumping and pressurize it.

2. Connect a pressure relief valve.

3. To collect mixture back in the tank, Start pumping still and apply heat to the still and M.C. as much as in bakeout. When the tank pressure is close to 385 Torr, use diffusion pump.

4. Collect residual mixture in the pipe line into the He3 pump.

5. Close the condenser and still valve.

6. Close all other valves.

7. Turn off cooling water to the diffusion pump.

8. Close the main valve of He gas cylinder.

9. Change of sample

1. Pump IVC. He leak check is recommended. Then put exchange gas into IVC.

2. Pump both condenser and still line.

3. Pump and flush the 1K pot three times and then pressurize 1K pot with helium gas (5 psi).

4. Pre-cool IVC with L.N2 for about 30 min.

\(^3\)Do not take cold trap out of L.N2 dewar before pumping it. When the cold trap is out, the pressure can shoot up.
5. Lower the insert slowly in the dewar. Do it slowly to save L.He.

6. Replace the 1K pot rubber hose with SS flexible hose.

7. Pump the SS hose section first and then pressurize it with He gas. Open 1K pot valve.

8. **Check if 1K pot is open!** 1)Stop pressurizing and see if the pressure goes down 2)Pump 1K pot and see if 1K pot resistance increases.(600-620Ω)

9. Connect condenser and still.

10. Pump gas handling lines. IF the colde trap is cold, it should be isolated with closing two valve.

11. IF the cold trap is at RT, dip it in the L.N2 dewar. Isolate the Still line by closeing the valve (right above the thermocouple gauge). Release a little amount of mixture from He3 pump into the cold trap to help it cool down.

12. The remaining steps are the same as below 5.4.