Three stage vacuum system for ultralow temperature installation

N. K. Das, J. Pradhan, Md. Z. A. Naser, B. Ch. Mandal, A. Roy, P. Kumar, C. Mallik and R.K. Bhandari

Variable Energy Cyclotron Centre, Department of Atomic Energy, 1 / AF, Bidhan Nagar, Kolkata- 700 064, India;
E-mail: nkdas@vecc.gov.in

Abstract. We use a three stage vacuum system for developing a dilution fridge at VECC, Kolkata. We aim at achieving a cooling power of 20µW at 100mK for various experiments especially in the field of condensed matter and nuclear physics. The system is essentially composed of four segments-bath cryostat, vacuum system, dilution insert and 3He circulation circuit. Requirement of vacuum system at different stages are different. The vacuum system for cryostat and for internal vacuum chamber located within the helium bath is a common turbo molecular pump backed by scroll pump as to maintain a vacuum ~10^-6mbar. For bringing down the temperature of the helium evaporator, we use a high throughput Roots pump backed by a dry pump. The pumping system for 3He distillation chamber (still) requires a high pumping speed, so a turbo drag pump backed by a scroll pump has been installed. As the fridge use precious 3He gas for operation, the entire system has been made to be absolutely leak proof with respect to the 3He gas.

1. Introduction
With a mission to build up a dilution refrigerator to obtain a cooling power of 20µW at a temperature of 100mK, we are using a bath cryostat with 25 litres of available LHe capacity with a hold time of 30hrs. The cryostat incorporates an internal vacuum chamber (IVC) which contain the dilution insert. The dilution insert is consisted of a helium evaporator, heat exchanger, 3He distillation chamber and a mixing chamber. The helium evaporator has a volume of 99cc and is made of OFE copper. Capacities of distillation chamber and mixing chamber are of 40cc and 10cc respectively. The schematic view of the system is shown in figure1. It operates at sub-atmospheric pressure and necessitates all subsystems and network of capillary interconnections truly leak proof. Vacuum of the system relies on three distinct stages-a common turbo-pumping station for cryostat and IVC, a root pump backed by dry pump is used for helium evaporator and a turbo molecular drag pump backed by scroll pump for 3He circulation.

IVC is housed within the helium bath of the cryostat with its top flange immersed in LHe. As such the leak tightness of the flange was ensured with the help of indium o-ring corner seal. The IVC is made of OFE copper with a height and diameter of 350mm and 130mm respectively. It is evacuated to about 10^-9torr using turbo molecular drag pump. IVC plays a significant role by way of thermally isolating the very low temperature system from the LHe bath. The evaporator which is used for precooling and condenses the circulating gas is again placed inside the IVC. The vapour pressure of the evaporator is reduced separately by a root pumping system (250m³/hr). 3He circulation unit pumps...
out the primarily $^3$He vapour from the $^3$He distillation chamber which contains a mixture of super fluid $^4$He and $^3$He. Since the distillation chamber maintains a temperature of 0.7K, the vapour pressure of $^3$He is about 1000 times higher than that of $^4$He. Therefore, the vapour above the super fluid will be essentially $^3$He.

2. Superfluid suppression
A common method of reducing film flow in dilution refrigerators is a knife-edge device. The principle behind the knife-edge film killer is that a superfluid film is reduced when it flows around a sharp bend. If the bend is sharp enough, the film does not flow past it. Unfortunately, there is a catch: the film evaporates where it stops, cooling the material of the knife-edge. As the knife-edge cools, some of the helium vapor condenses on the evaporator. This newly condensed, somewhat reduced, film then continues to flow downstream.

3. Evaporator pumping system
This is the continuously filled evaporator (1K pot) design and tested in the laboratory and found to offer a temperature less than 1.0K by withdrawing vapour from the evaporator. In order to minimize the heat load and to prevent film creep across the pumping tube, size optimization of the pumping line and pump-out port has been performed. Five nos thermal Baffles-are used along the pumping line as thermal barriers to radiative heat load.

3.1. Basis to choose the pump
To achieve the available cooling power of 10 mW@1.0K, the required steady state molar flow rate that has to be pumped out by the pump is calculated to be 250 μmols/sec. For the minimum ultimate evaporator temperature of 1K the vapour pressure at the pot has to be maintained 0.1 torr. Initially if we consider the pressure drop along the pumping line be negligible, then from the ideal gas equation

$$P\dot{V} = nRT$$

where P is the pressure at the pumping line at the top of the cryostat, T is the room temperature (300K), $\dot{n}$ and $\dot{V}$ are the molar flow rate and the pumping speed at the top of the
cryostat. For \( P = 0.1 \) Torr, the pumping speed is calculated as 168 m\(^3/hr\). To minimize the vibration arises due to the pump the pump is placed few meter away from the cryostat. Considering this the pump of pumping speed 250 m\(^3/hr\) is chosen.

Fig. 2. Temperature and pressure distribution along the pumping line of the evaporator (1K pot).

4. Optimization of the pumping line inside the cryostat

The geometry of the pumping lines are chosen such that the conduction heat load to the 1K pot as well as to the LHe bath are minimum and the pressure drop along the pumping line is as minimum as possible. The wall thickness of the pumping section connected to the evaporator must thin, small diameter to keep the cross sectional area for heat conduction as low as possible\(^{[1]}\). The superfluid that climbs up the pumping line should evaporate before reaching the pumping section that have larger diameter to pump out the more vapour emerging out of the superfluid evaporation as well as normal fluid evaporation. With a view to make an optimal balance between thermal conduction load (\( Q \propto r \)) from and viscous flow conductance (\( C \propto D^4 \)) of the pumping line, it has been designed in three sections having different diameters.

Figure 3: Mass flow rate through evaporator pumping lines for different heater settings.
The pumping line consists of mainly three sections with different length and diameter in order to make balance between the thermal load and pumping speed. The conductance of the piping system is primarily depends on the sizing of orifice diameter at the entry of 1K pot. The ANSYS FLOTRAN™ CFD module was used to obtain pressure and temperature distribution for sizing of the piping lines. We have obtained ultimate temperature of less than 1K and cooling power of ~17milliwatt at 1.2K continuously. We could control the temperature by fiddling with needle valve from outside. The absolute pressure was measured at room temperature near to the cryostat. The standard chart provides the vapour pressure inside the pot from its temperature. The above figure shows the variation of mass flow rate obtained from the heater power with the pressure drop along the lines. The slope of curve which is proportional to the conductance of pipe system is same as the estimated value of 30m3/hr. The vertical shift of the measured curve from the calculated value is possibly due to error in our meter reading, which is not accurate enough to monitor the small pressure variations.

![Diagram of temperature contour](image)

Fig. 4. Temperature contour of the different sections of the evaporator (1 K pot) pumping system.

### 5. $^3$He Circulation Circuit

An important feature of the refrigerator is the $^3$He circulation system. The cooling power of dilution fridge is dependent on the $^3$He circulation rate. As only modest pressures are required to maintain the cold of the Still, much higher circulation rates are available by using a turbo-drag pump to circulate $^3$He rather than a diffusion pump. The turbo-drag pump also requires a comparatively small backing pump than that of a diffusion pump. The pumping system chosen for this application is a turbo-drag pump (Edwards; model No: EXD556H) backed by helium sealed scroll pump (Edwards; model No: XDS35i). The maximum $^3$He circulation rate available with this combination of pumps was 30µmoles/s. As a way of minimizing the heat conduction through the pumping line, the diameter of the line has been incrementally increased towards the 300K. Length of 150 mm(OD:9.5 mm) from still followed by 1 inch pipe of length 250 mm and 200 mm of 2” diameter pipe have been used to make the line. Even though the evaporating $^3$He in the Still is pumped through 1mtr flexible line after 300K,
the Still pressure has been estimated to be about 0.1 torr. The total conductance of the pumping line is estimated of 15 m³/hr. This high circulation rate minimises the cool-down time of the refrigerator.

Fig. 5. Variation of vapour pressure with still power

In order to circulate and get the continuous cooling in the mixing chamber of the dilution fridge, the vapour above the STILL is required to pump out continuously. The vapour constitute mainly of ³He, which at the temperature of STILL ~ 0.7K is about 1000 times more vapor pressure than ⁴He. The heater is provided around the STILL to maintain the required flow and its vapour pressure variation with heater power is depicted in figure 5 above. The pumping lines for the STILL are designed taking into account the compromise between reducing the thermal load and increasing pumping speed[2]. The orifice of diameter 2mm is provided at the entry to restrict the super fluid film flow, which basically limits the conductance of the pumping lines.

Fig. 6. Pressure and temperature distribution along the still pumping lines.

The pressure and temperature distribution along the three sections of pumping lines is shown. The lines are anchored at suitable positions at different temperatures for reducing the heat load at lower temperature.
6. Leak testing of the vacuum system
Vacuum and pressure leak test were carried out for all joints, seals and welding points. Convenient and reliable sealing in the IVC and helium evaporator and 300K flange were made from indium o-ring to avoid the spillage of liquid helium. Indium material used is ultra-pure (99.9 minimum purity) to prevent hardening of the material at sub-Kelvin temperatures, as well as to restrict impurities of elements with low vapour-pressure. The indium seal joints have undergone for the helium leak test under following conditions-room temperature leak test of the indium seal joints, test after repeated thermal shock at liquid nitrogen temperature, helium leak test of indium seal at liquid nitrogen temperature in flow condition. The schematic view of the leak-testing set up is shown in figure 7. The average measured leak rate of all seals was ~4.0×10⁻¹⁰mbar-l/s by helium leak detector.

Fig. 7. Schematic diagram for leak testing set up.

7. Conclusion
Three sets of pumping system have been optimally selected to run the dilution refrigerator. Sizing of the pumping line & capacities have been taken in to account to satisfy the requirement of the system. Leak testing of all the joints seals and the pumping line have been thoroughly carried out.

Reference
[1] DeLong L E et.al. 1971 Rev. Sci. Instr. 42, Number 1, pp 147-150
[2] Richardson L J et.al. 1978 Cryogenics February pp 109-111