Small-Size Dilution Cryostat for Rapid Cooling

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Abstract

A small test dilution cryostat has been constructed. Reliable operation with base temperature of 50 mK was obtained. Design considerations and performance are discussed. Cool down from room temperature to 100 mK takes 3 – 5 hours.

1 Introduction

Ultralow temperature experiments are usually performed in large cryostats for several reasons: efficient heat exchangers of dilution cryostat with large cooling power require some volume and nuclear cooling with copper demands large magnetic fields of $\sim$ 6 T and a big nuclear stage. Such large cryostats have a long cool down time and require carefull testing of vacuum leaks and gas flow within the cryostat. Recently there has been increasing interest in small dilution cryostats capable of reaching a base temperature of 10 – 50 mK [1, 2, 3]. These have fast turn around time and are well suited for testing different devices or for checking out equipment for experiments which will later be conducted in a big nuclear demagnetisation cryostat. It is likely that most practical problems will be uncovered by such testing in the small dilution machine. For instance, measured signals and thermal noise are on a more realistic scale than in more simple tests at 1.5 – 4 K in liquid ^{4}He dewar.

2 Operating principle of a dilution cryostat

A mixture of ^{3}He and ^{4}He liquids phase separates into two parts below 0.87 K [4, 5, 6], Fig. 1. The upper volume consists of the ^{3}He rich phase and the lower volume of a dilute solution of ^{3}He in superfluid ^{4}He. The concentration of ^{3}He in the upper volume approaches unity when T $\rightarrow$ 0 K. In the diluted solution the concentration of ^{3}He stays finite and approaches 6% at zero temperature. When ^{3}He atoms move from the ^{3}He rich phase to the diluted phase cooling is observed. The cooling power is due to the difference in the enthalpy of the diluted and concentrated ^{3}He phases

$$\dot{Q}_M = \dot{n}_3(96T_M^2 - 12T_N^2), \tag{1}$$

where $\dot{n}_3$ is the amount of ^{3}He atoms crossing the phase boundary in mol/s, T_M the mixing chamber temperature, and T_N the temperature of the incoming ^{3}He. For zero heat load the minimum temperature is determined by the temperature of the incoming ^{3}He stream $T_M \approx T_N/2.8$.

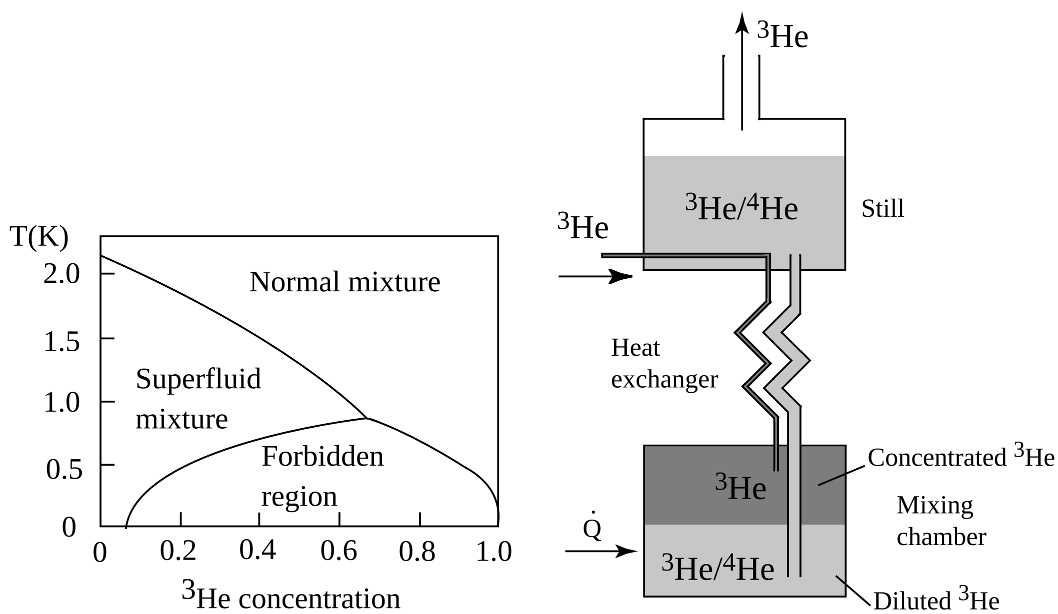

Figure 1: *Left:* Phase diagram of ^{3}He/^{4}He mixtures. The molar concentration of ^{3}He in the mixture $n_3/(n_3 + n_4)$ is plotted on the horizontal axis. Below 0.87 K the mixture phase separates into two different volumes: a ^{3}He rich phase floats on top of a diluted ^{3}He/^{4}He phase. At zero temperature the concentration of ^{3}He in the upper volume approaches unity while for the lower volume it becomes 6.4 %. *Right:* Principle of a dilution cryostat. The cooling is produced in the mixing chamber where ^{3}He atoms move from the ^{3}He rich phase to the diluted ^{3}He/^{4}He phase. The ^{3}He flow is maintained by evaporating the ^{3}He from the diluted solution in the still. The pumped gas is returned back into the cryostat and condensed to liquid before it enters the heat exchanger in the still.

In a practical dilution cryostat the phase boundary between the diluted ^{3}He and concentrated ^{3}He is located in the mixing chamber. The incoming ^{3}He is cooled in a heat exchanger. A second vessel above the mixing chamber, the still, is used for evaporating ^{3}He. The pumped gas is fed back to the cryostat and condensed to liquid at the ^{4}He evaporation cooler, the ^{4}He pot.

3 Design considerations

The cryostat should be easy to use with fast cool down time. It should have a modular structure to allow easy modification for different experiments. There should be space for many shielded wires, GaAs MESFET amplifier, DC SQUID with its shields and for an experiment below the mixing chamber. A 1 K plate with a ^{4}He pot is needed. It serves as the first thermal anchor for the wires inside the cryostat. It also provides a fixed operating temperature for the DC SQUID when the liquid ^{4}He level in the dewar is already below the flange of the vacuum jacket. The vacuum can and pot flanges should have the provisions to fix different accessories on them.

3.1 ^{4}He pot - design and performance

The pot design is fairly simple: it is a copper vessel soldered on the pot flange, Fig. 3. A 1.6 mm diameter CuNi tube connects the pot volume to a larger pumping tube. It works as a superfluid He-II film stopper at the same time. In the pot the returning ^{3}He is condensed to liquid in a coiled copper tube. The pot is continuously filled with a syphone extending to the liquid helium bath outside. The temperature of the pot is measured with a RuO_2 chip resistor of nominal value of 1 kΩ (type RCWPM-575 from Dale Electronics).

The main optimization parameter for the pot is the flow impedance from the liquid helium bath. The pot should not run empty too easily and does not need to maintain very low temperature. Thus the impedance can be of fairly low value, 10 - 20 cm of 0.1 mm inner diameter CuNi tube is usually sufficient. The tall shape of the pot gives some reserve to sudden heat loads (like in starting the condensation of mixture or circulation in the dilution machine). In stable operation of the pot the liquid level inside it is high, close to the 1.6 mm CuNi pumping tube. This can be seen in the small oscillations in the base temperature of the pot: When the liquid level reaches small pumping tube, the evaporating and cooling power are suddenly decreased and the pot warms up. It cools down again when the liquid level drops so that there is a larger evaporating area. This was tested with different impedances. First a small impedance was used and the oscillations in the pot temperature started immendiately when the pumping was started. The reason for this was the rapid filling of the pot. With a larger impedance the temperature was stable in the beginning and started oscillating between 1.68 - 1.70 kΩ after 30 minutes of pumping.

The pot cooling power was tested with a heater, Fig. 2. First the pot was filled with liquid ^{4}He. The heat load was gradually increased and a small drop in the thermometer resistance was observed. The heating was kept constant for five minutes. After the last point with 50 mW of heating the pot ran empty. This was seen as a sudden drop in the thermometer resistance to 1250 Ω. Thus with the gradually increasing heating the pot was empty in 30 minutes. When the heating was decreased the thermometer resistor followed a different curve and the pot stayed at a relatively high temperature. Below 10 mW heating it started to fill with liquid again.

3.2 Still

The still is pumped via a 8 mm diameter tube. There is a sharp knife-edge orifice in the pumping channel to restrict the ^{4}He superfluid film from creeping up to a high temperature point in the pumping tube and evaporating there. At the knife edge there is a large gradient in the pressure. Part of the ^{4}He film will evaporate there. The rest of the film flow is reduced due to the small surface area of the knife-edge. The ^{4}He circulation is a problem because it does not participate in the cooling of

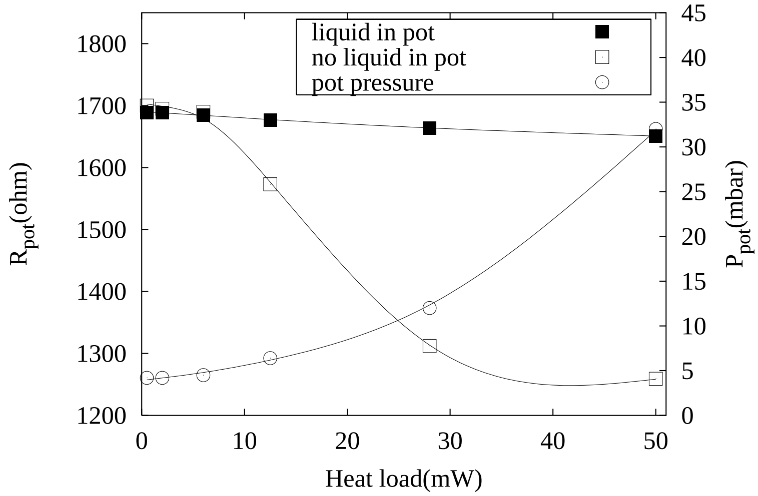

Figure 2: The pot temperature at different heat loads.

the dilution refrigerator but loads the pumping system [5]. The restriction is in practice, however, a sufficient precaution for a small dilution machine. The still has an indium sealed bottom. To this bottom part four copper feedthroughs have been hardsoldered. It is easy to use these feedthroughs by soldering a CuNi tube into them with ordinary soft solder. The CuNi tube contains either the circulating ^{3}He or wires going into the still. The wires are sealed to this tube with the standard method by using a feedthrough molded from Stycast 1266 epoxy [4, 6]. The temperature of the still is measured with a RuO_2 resistor of nominal value 1 kΩ. The heater is made from twisted CuNi wire wound inside on the bottom plate of the still. The incoming ^{3}He is thermalised to the still temperature with a copper block fixed with a screw to the bottom. A second flow impedance (5 - 10 cm of 0.1 mm inner diameter CuNi tube) before the continuous heat exchanger ensures that no condensation of re-evaporated ^{3}He happens in the still, or worse, in the heat exchanger.

3.3 Continuous heat exchanher

The continuous heat exchanger is made from 1.6 mm diameter CuNi tube with a PTFE tube of 1 mm diameter inside it. The connection between the second flow impedance and the Teflon tube is sealed with vacuum grease. The thermal contraction of the Teflon helps in making this joint leak tight. On the otherhand, even if there is a small leak, it should not deteriorate the performance too much. The plastic has a small density compared to the usual metallic heat exchangers. Thus the phonon mismatch, giving rise to Kapiza boundary resistance [7] for heat flow between solid and liquid, is smaller. The CuNi tube is wrapped on a Teflon frame mechanically connecting the still and the mixing chamber. This frame gives a fairly large, of order of 1 μW, heat leak to the mixing chamber.

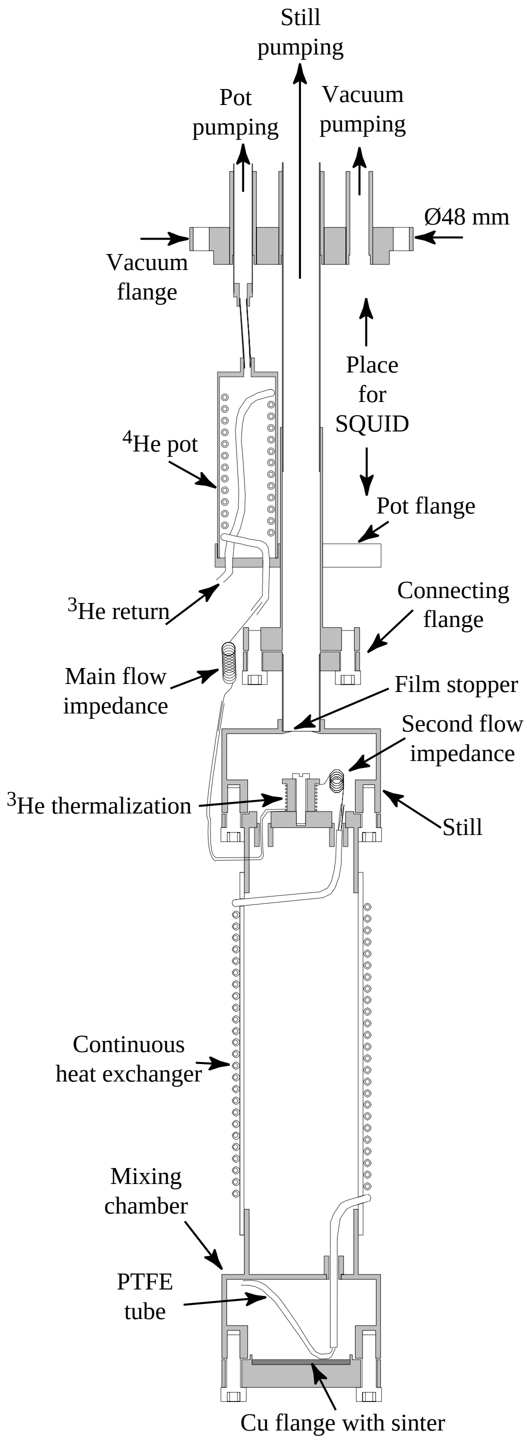

Figure 3: Construction of the dilution cryostat. The vacuum flange on the top has three pumping tubes for vacuum, pot and still. The ^{4}He evaporation cooler or pot gives the 1.5 K temperature that is needed for condensing incoming ^{3}He (and the ^{4}He in the initial filling) of the dilution unit. The main flow impedance is a 20 cm long, 0.1 mm inner diameter CuNi tube. The still pumping tube is connected with a indium sealed flange to the still. Thus the unit can be easily removed. The still can also be opened easily. A teflon frame connects mechanically still to mixing chamber. A simple continuous heat exchanger with a CuNi outer and PTFE inner tube is used. The experiment is fixed to the mixing chamber flange.

3.4 Mixing chamber

The mixing chamber has the same volume as the still. It is made from stainless steel AISI 316L with a bottom flange made from copper and sealed with an indium O-ring joint. The Cu-flange has silver powder sintered on it. There are threads on the Cu-flange for fixing thermometers, heaters and the experiment. The Teflon tube that carries the incoming ^{3}He goes to the concentrated part of the mixture. The diluted fraction of the solution is pumped through the annular volume between the Teflon tube and the CuNi tube.

4 Cooldown from room temperature

The vacuum jacket of the cryostat is sealed with an indium O-ring joint. It is pumped through the vacuum pumping tube. Usually the valves to the dilution machine are kept closed when the dilution machine is warm. Thus there is no need to pump the dilution machine. The same is true for the ^{4}He pot: After the previous cooldown it is filled with an overpressure of gaseous ^{4}He. The overpressure is leaking slowly from the pot through the syphone. No air or moisture will be flowing to the pot which would block the flow impedance capillary during cool down. Before cooldown the pot is pressurised with 1.5 atm of helium. Thus there will be a small overpressure in the pot during the cool down to the liquid nitrogen temperature.

The cryostat is inserted into liquid nitrogen vessel for precooling. By monitoring the vacuum pressure it is possible to see if there are any leaks. The pressure should decrease monotonically. A fairly accurate pressure gauge with digital display is useful in monitoring such small changes in the pressure. Only if a leak is observed is it necessary to use a leak detector to determine where the leak is. Most leaks are observed already at LN_2 temperature. Heat exchange gas is necessary to cool the inner parts of the cryostat. The gas can be either air, neon or ^{4}He. The precooling with 1 – 5 mbar of helium is fast and takes about 10 – 15 minutes. Neon is used if there is need for leak detection of the dilution unit or experimental cell at LHe temperature. Precooling with air is slow and it is seldomly used.

After the cryostat has cooled to LN_2 temperature it is moved into the ^{4}He storage dewar. The helium heat exchange gas works also at LHe temperature and it is not necessary to pump it finally. If the amount of heat exchange gas was right in the begining it will be effectively cryopumped by the cold walls of the cryostat. Thus it does not necessarily need to be pumped away as the cryostat can operate with smaller than $5 \cdot 10^{-3}$ mbar pressure of helium in the vacuum jacket. The pot fills automatically from the liquid ^{4}He bath when the temperature goes down. Its operation is started by pumping through the pot pumping tube. The pot operation temperature of about 1.5 K is reached almost immediately. If there are any problems with pot operation they become now visible: Too low a temperature means that the syphone or impedance has been partially blocked by air and a superleak in the pot will be seen clearly as an increase in the pressure of the vacuum jacket.

When the proper ^{4}He pot operation has been confirmed, condensation of the ^{3}He/^{4}He-mixture into the dilution unit can be started. This is done simply by fully opening tank volume into the still pumping tube. The pot thermometer resistor will collapse to a lower value in about 15 – 30 minutes indicating that the pot has run empty of liquid ^{4}He. When it recovers in 1 – 2 hours time most of the condensation has taken place and further condensation proceeds very slowly. At this point it is better to start the circulation and to condense the rest of the mixture to the dilution unit while circulating gas through the refrigerator. The still valve is opened slowly and the still is pumped for 30 minutes before the ^{3}He return line to the cryostat is opened. This way one makes sure that all the air goes into the LN_2 trap and does not block the cold ^{3}He return line. The needle valve from the storage tank is opened to the still pumping tube so that the incoming flow of ^{3}He does not dry the pot with too much heat load. Usually the flow is limited to below 100 μmol/s. When all the gas has been added to the circulation and stable operation has been reached, then the still heater is switched on. The mixing chamber starts to cool down rapidly reaching 100 mK in about one hour.

5 Performance of the dilution cryostat

The minimum temperature in the mixing chamber, about 50 mK, was reached with about 0.7 mW heating power to the still. With larger heating the temperature started oscillating with a period of 15 - 30 minutes. These oscillations are probably due to emptying of the still; with too much heating power the mixture in the still evaporates. The area of the heat exchanger tube connecting the still and mixing chamber is much less than the cross section area of the still. Thus, less ^{3}He evaporates and circulation decreases. When the extra ^{3}He comes back to the mixing chamber, the still is again filled with mixture and the cycle repeats itself.

The cooling power can be measured by supplying constant heat to the mixing chamber and waiting for the temperature to stabilize, Fig. 4. A heater resistor made from 100 μm CuNi wire is used for heating. The circulation was $\dot{n}_3 = 35$ μmol/s and cooling power at 100 mK about 10 μW. The minimum temperature is a little uncertain since the Ge-resistor (Lake Shore GR-200A-50) saturated at 1.3 MΩ. The thermometer is easily overheated at such high values and may indicate a higher temperature than the mixing chamber. It was calibrated against a same type factory calibrated Ge-resistor. The estimated heat leak through the teflon frame from the still to the mixing chamber is about 1.6 μW. From the cooling power measurement, a 3.8 μW heat leak to the mixing chamber was obtained.

6 Conclusions

A small dilution refrigerator has been constructed which fits inside a liquid He container with a 50 mm neck and achieves reliably a base temperature of 50 mK and a cooling power of 10 μW at 100 mK. The cool down from room temperature takes 3 - 5 hours, depending on how succesful each precooling step is. The low temperature performance of the cryostat could still be improved with better heat exchangers, e.g. with the "bellows" type step heat exchanger made from Kapton foil [7]. On the other hand a construction with soldered joints has better reliability in repeated thermal cycling than one with large plastic parts. With less ^{3}He in the mixture the dilution unit should cool down faster [4]. The operation is sensitive to the condensing pressure and the amount of gas in the room temperature of the pumping system: too high a pressure means that the phase boundary is not in the mixing chamber since too much ^{3}He is in the back of the pump. The space between the still and mixing

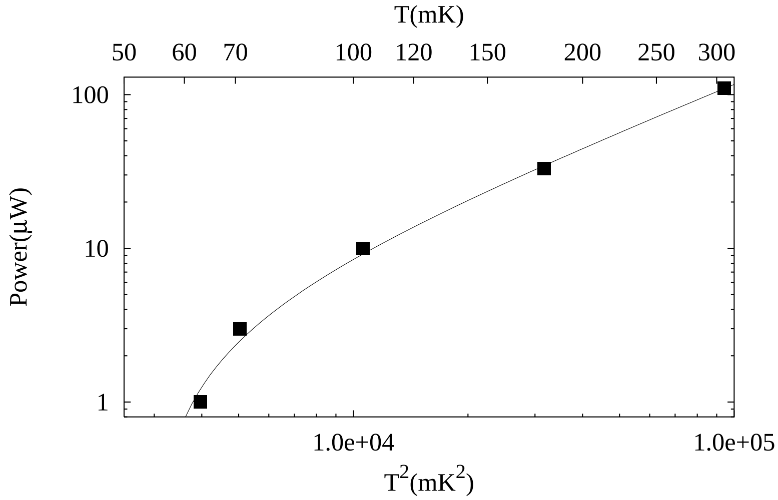

Figure 4: The mixing chamber cooling power. The heating power is plotted on the vertical axis. Below 1 μW, the heating is dominated by an external heat leak. A lower base temperature could be obtained by reducing this heat leak. The line represents a fitted $\dot{Q} \propto T^2$ dependence.

chamber is now not used. With a better design of the mixing chamber this space could be used for experiments or a step heat exchanger could be placed there.

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