

4.2 Elements of cryogenics

Helium is the only element that remains liquid when cooled to the lowest possible temperatures (well below 1 mK) under atmospheric pressure. It is therefore the prime candidate as a refrigeration medium for temperatures below the condensation temperature of nitrogen (77 K). The vast majority of mesoscopic transport experiments are performed in this tempera-

ture range. The latent heat that has to be paid when liquid helium is evaporated is the cooling power made use of in helium cryogenics. Continuous evaporation of liquid is possible by pumping off the vapor pressure. Therefore, we will look at the properties of liquid helium (LHe), as well as cryostats, the devices used to establish low temperatures.

4.2.1 Properties of liquid helium

The physics of LHe is extremely interesting and rich, and experimentalists working on transport in nanostructures will almost inevitably get in contact with its unusual properties.

Helium comes in two isotopes, the boson ^4He and the fermion ^3He . The mono-isotopic liquids have therefore very few properties in common. As a liquid is cooled, kinetic energy is taken away from the atoms. At the condensation temperature, the attractive interatomic van der Waals forces start to dominate in any liquid other than LHe, and the crystallization sets in. He is the only element for which the van der Waals force is smaller than the kinetic energy of the atoms due to zero-point fluctuations. The van der Waals forces in He are particularly weak since the atoms have no dipole moment. On the other hand, the zero-point fluctuation energy is particularly large, due to the small atomic mass. Only by applying a pressure above ≈ 30 bars, the atoms are pressed sufficiently close together such that crystallization sets in.

So much as for the common properties of ^3He and ^4He . We now look at some properties of the pure isotopic liquids, before we turn to the interesting issue of $^3\text{He}/^4\text{He}$ mixtures.

Some properties of pure ^4He .

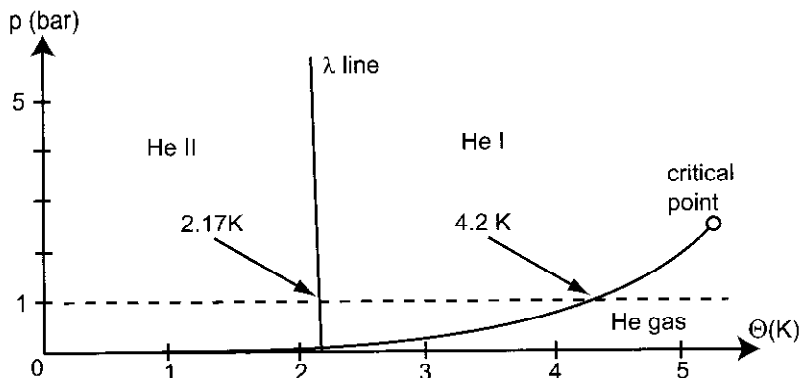


Figure 4.18: Phase diagram of the ^4He .

Fig. 4.18 shows the phase diagram of ^4He . Under atmospheric pressure, it liquifies at $\Theta = 4.2$ K. The density of the liquid $\rho(\text{L}^4\text{He}) = 125 \text{ kg/m}^3$. The vapor pressure drops approximately exponentially as LHe gets colder, and reaches 1 mbar at $\Theta = 1.2$ K. As we cool the liquid, we cross the λ line at some temperature, which for atmospheric pressure happens at $\Theta_\lambda = 2.17$ K, also known as the λ point. The λ point got its name from the specific heat as a function of Θ around this transition, a function that looks like this omnipresent Greek letter. For $\Theta > 2.17$ K, ^4He behaves just like any ordinary liquid. As we lower Θ and cross the λ point, ^4He undergoes a phase transition and develops highly remarkable properties. L ^4He in this phase is often referred to as HeII. In fact, the phase transition at the λ point can be modelled as a Bose-Einstein condensation, i.e., the condensation of a boson gas. Within such a model, the ^4He above the λ point is described as a gas, which is not a bad approximation, considering the weak interactions. At $\Theta = 0$, on the other hand, all atoms of HeII are in the ground state. At higher temperatures, the energy levels in a Bose-Einstein condensate (BEC) are occupied according to the Bose-Einstein distribution function

$$f_{BE}(E, \Theta) = \frac{1}{e^{(E-\mu)/k_B\Theta} - 1}$$

A pure BEC, however, cannot explain the observed behavior of HeII. Rather, [London1938] proposed a two-liquid model, which treats HeII as a mixture of a normal fluid and a superfluid, which interpenetrate on a microscopic length scale, similar to the electronic state in a type II superconductor. The normal fluid behaves just like ^4He above the λ point. In particular, it has a non-vanishing entropy and viscosity. The superfluid, on the other hand, has zero entropy and viscosity, which means, for example, that there is no flow resistivity. Furthermore the

thermal conductivity of the superfluid is infinitely large. How the composition of HeII changes with temperature has been measured by [Andronikashvili1946]. In this seminal experiment, a torsion pendulum made of a stack of thin disks was immersed in HeII, and the damping of the oscillation was measured as a function of temperature. Since the normal fluid is viscous, it adds to the moment of inertia of the system via the law of Hagen-Poiseuille, while the superfluid does not. The measured composition of HeII is shown schematically in Fig. 4.19. As the temperature is lowered, the normal fluid fraction rapidly vanishes and an almost pure superfluid remains for $\Theta \lesssim 0.7$ K. This two-component mixture has some unique properties

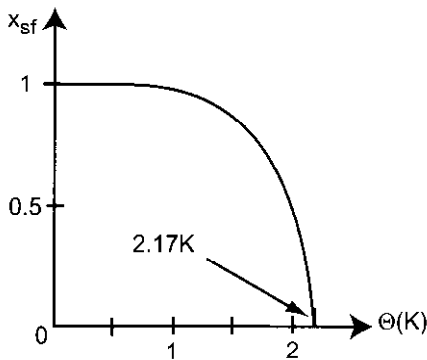


Figure 4.19: Superfluid fraction x_{sf} of HeII as a function of temperature. After [Andronikashvili1946].

we should know, in order to appreciate its behavior in cryogenic equipment.

1. Absence of bubbling.

If we heat a conventional liquid, it starts bubbling, since the liquid evaporates at some random spot, and the gas bubble rises to the surface. In HeII, the thermal conductivity is very large, and evaporation takes place at the surface only. Hence, HeII is perfectly quiet, even if it boils off. In a simple picture, we can understand the extremely high thermal conductivity as follows. Imagine we connect heat reservoirs to both ends of a tube filled with HeII. At the end with higher temperature, superfluid is transformed into normal fluid, with a final ratio in accordance with Fig. 4.19. The heat is transferred to the low-temperature end by normal fluid convection. Here, the normal fluid is re-transformed into superfluid. Since the superfluid carries no heat (its entropy is zero), all the heat is thereby absorbed by the heat sink. The heat transfer is therefore very efficient. The heat conductivity is further increased by the extremely low viscosity of HeII, which means there is vanishingly small friction during the convection process.

2. HeII osmosis.

Consider two chambers filled with HeII, connected to each other by a *superleak*, i.e., a connection only permeable for superfluid helium, see Fig. 4.20. Such connections can be made by extremely fine capillaries, or by tubes stuffed with powder. This setup immediately reminds us of an osmotic pressure cell, with the semipermeable membrane being

the superleak, the solvent being the superfluid, and the normal fluid component starting as the solute. Recall that in osmosis, the solute can be thought of a gas, and that the osmotic pressure evolves due to the tendency of the solvent to equalize the concentrations in both chambers. As we heat HeII in one chamber, the fraction of normal fluid increases, and superfluid will enter this chamber, in order to dilute it. Consequently, a pressure difference is built up. In equilibrium, the hydrostatic pressure will compensate the osmotic pressure, and the surface positions in the two chambers will differ by Δh .

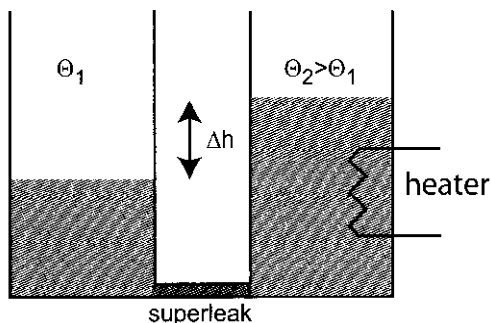


Figure 4.20: Sketch of a HeII osmotic cell.

3. Superfluid film creeping.

HeII tends to creep over any wall of reasonable height, as long as its temperature stays below the λ point. Therefore, containers filled with HeII to different heights will equilibrate their surface levels, see Fig. 4.21. This effect has its origin in the extreme adhesion of HeII to surfaces. Within the framework of liquid-solid interfaces, this is known as "complete wetting". Since the shape of the liquid surface is determined by the condition that the tangential forces vanish, this effect occurs for $-\sigma_{ls} > \sigma_{gl}$, where σ_{ls} (σ_{gl}) denote the liquid-solid (gas-liquid) interface tension.

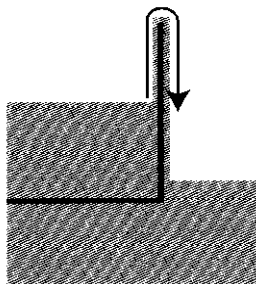


Figure 4.21: Superfluid film creeping across a wall.

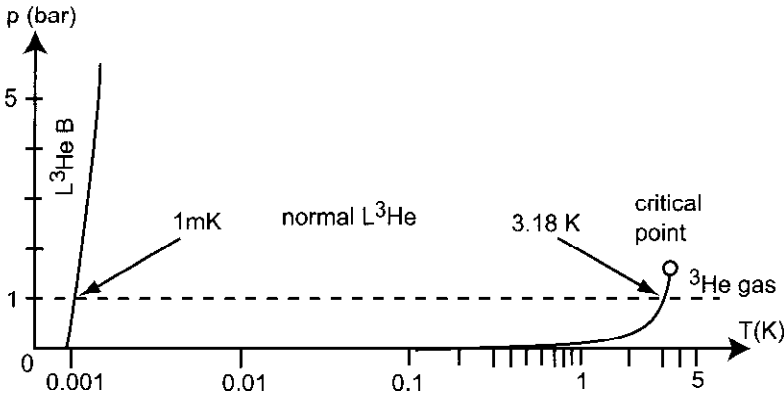


Figure 4.22: Phase diagram of ${}^3\text{He}$.

Some properties of pure ${}^3\text{He}$.

In Fig. 4.22, the phase diagram of ${}^3\text{He}$ is sketched. For our purposes, the additional phases occurring at extremely low temperatures below 2 mK are irrelevant.⁴ L ${}^3\text{He}$ has a density of $\rho_{{}^3\text{He}} = 59 \text{ kg/m}^3$. Under atmospheric pressure, it liquifies at $\Theta = 3.19 \text{ K}$. This boiling point is about 1 K below that one of ${}^4\text{He}$, which can be easily understood, since its mass is smaller, and thus the atoms have a larger average velocity at the same temperature. Consequently, the vapor pressure is also higher at identical temperatures. It drops to 10^{-3} mbars at about $\Theta = 270 \text{ mK}$. ${}^3\text{He}$ atoms are fermions, and the liquid can be approximated by a Fermi gas, with many analogies to electron gases.

Question 4.1: Calculate the Fermi energy of ${}^3\text{He}$!

Within the Fermi liquid picture, we can imagine that each ${}^3\text{He}$ is surrounded by a screening cloud, which results in quasiparticles with an effective mass given by the interactions. At atmospheric pressure, $m^*({}^3\text{He}) \approx 3m({}^3\text{He})$. For practical cryogenic purposes, ${}^3\text{He}$ behaves as an ordinary liquid.

A further important point concerning ${}^3\text{He}$ is its near-to complete natural absence on earth. It can be generated by nuclear reactions, and is consequently extremely expensive. Therefore, all ${}^3\text{He}$ cryostats keep it in a closed cycle.

The ${}^3\text{He}/{}^4\text{He}$ mixture.

Let us first look at the phase diagram of this mixture, Fig. 4.23. For $\Theta > 860 \text{ mK}$, nothing spectacular happens. The main effect of the ${}^3\text{He}$ is to reduce the λ point of the homogeneous mixture. Below the λ -line, ${}^3\text{He}$ dissolved in HeII can be just thought of an additional fraction

⁴For $\Theta < 2 \text{ mK}$, the ${}^3\text{He}$ atoms form Cooper pairs and undergo a Bose-Einstein condensation into superfluid ${}^3\text{He}$. Further phases exist at high pressures.

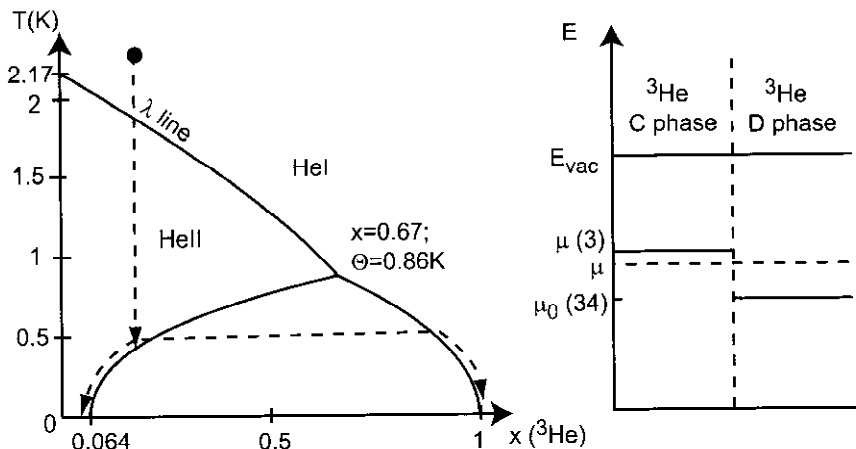


Figure 4.23: Left: phase diagram of the ${}^3\text{He}/{}^4\text{He}$ mixture vs. ${}^3\text{He}$ concentration x and temperature Θ . The tricritical point is at $x = 0.67$ and $\Theta = 860$ mK. At lower temperatures, the mixture segregates into a ${}^3\text{He}$ -concentrated phase (the C phase) and a ${}^3\text{He}$ dilute (D) phase. Right: sketch of the chemical potential of the two phases at $\Theta = 0$.

of the normal fluid component. For temperatures below 860 mK, a remarkable phase separation into a ${}^3\text{He}$ -poor phase (called the *dilute phase* - D phase in the following) and a ${}^3\text{He}$ -rich phase (the *concentrated* - C phase) takes place. At these temperatures, the pure HeII is almost completely superfluid, and the dissolved ${}^3\text{He}$ forms a normal fluid component.

A qualitative understanding of the phase separation can be obtained by recalling that ${}^3\text{He}$ is a Fermi liquid, while ${}^4\text{He}$ in this regime is a Bose-Einstein condensate. The ${}^3\text{He}$ dissolved in ${}^4\text{He}$ can be thought of a dilute Fermi gas with an effective mass given by the interaction between the ${}^3\text{He}$ atoms and the surrounding ${}^4\text{He}$, which is $m^*({}^3\text{He in } {}^4\text{He}) \approx 2.4m({}^3\text{He})$. Since superfluid ${}^4\text{He}$ has zero viscosity, the ${}^3\text{He}$ atoms can move around without friction, once the ${}^3\text{He} - {}^4\text{He}$ interaction is included in the effective mass. ${}^3\text{He}$ can be regarded as a Fermi gas as well. We just have to establish the conditions for which the chemical potentials of the C phase and the D phase are identical. Here, the superfluid ${}^4\text{He}$ plays no role, as all these atoms are in the ground state. The problem somewhat resembles the alignment of chemical potentials at interfaces discussed in the previous chapter. Here, the common energy level is again the vacuum level, i.e., the energy of a ${}^3\text{He}$ atom at rest in the vacuum. The chemical potential $\mu(3)$ of the C phase is somewhat higher than that one ($\mu_0(34)$) of a single ${}^3\text{He}$ atom in ${}^4\text{He}$, which can be understood by the fact that the (attractive) van der Waals forces are slightly larger in ${}^4\text{He}$, since the average separation of the atoms is smaller. Hence, ${}^3\text{He}$ atoms will go into ${}^4\text{He}$ until the chemical potentials have aligned. This is the reason why even at $\Theta = 0$, the D phase contains still 6.4% of ${}^3\text{He}$ atoms. Note that it is energetically unfavorable for ${}^4\text{He}$ atoms to reside in the C phase.

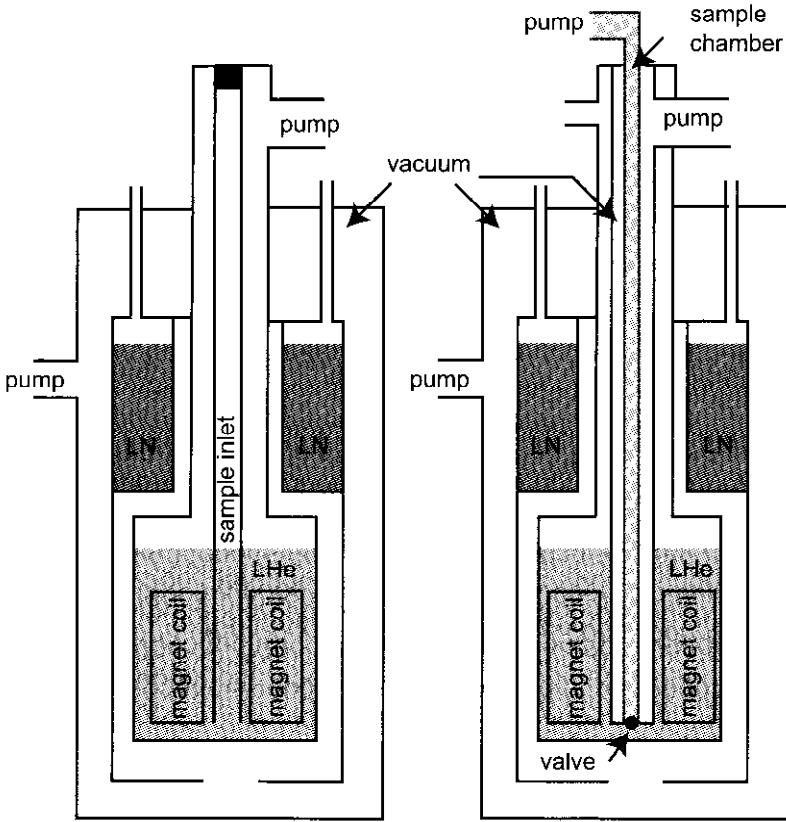


Figure 4.24: Sketch of a ^4He bath cryostat (left) and a ^4He gas flow cryostat (right).

4.2.2 Helium cryostats

Helium cryostats can be classified according to the kind of helium mixture for which they are designed. Occasionally, liquid nitrogen cryostats are used as well, for temperatures between 77 K and larger. However, from our discussion of the ^4He cryostat, their design should be pretty obvious. We begin with the “high-temperature” helium cryostats.

^4He cryostats

Helium has a small latent heat, which means it boils off easily. Therefore, the LHe cryostat has to be thermally decoupled from the environment. This is achieved by several means. Separating the He vessel from the outer world by a vacuum avoids heating via convection. Second, the LHe container is made of a material with a poor thermal conductivity, such as glass or stainless steel. Finally, the thermal radiation from the environment is shielded by surrounding the LHe vessel with liquid nitrogen, in order to reduce the temperature of the blackbody radiation that hits the He dewar. Alternatively, it is possible to wrap the dewar in

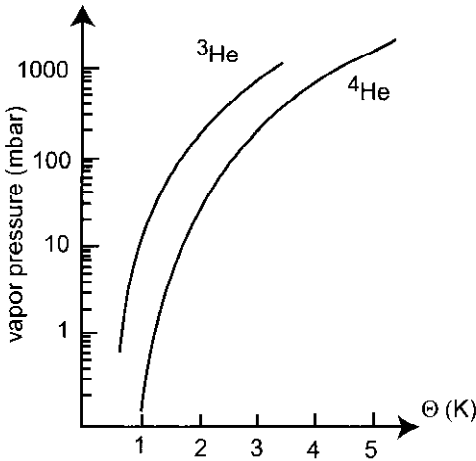


Figure 4.25: Vapor pressure of ^3He and ^4He .

”super-insulating” foil, which is a multilayer of insulating foil, where each layer is coated with a metal on one side. Examples of L^4He cryostats are shown in Fig. 4.24. In a *bath cryostat*, the sample is simply immersed in the LHe. The liquid, and with it the sample, can be cooled by pumping away the He vapor. This causes LHe to evaporate, which costs the latent heat and thus cools the liquid. The pumping speed and the incoming heat flux essentially determine the lowest possible temperature. To be somewhat more quantitative, recall the Clausius-Clapeyron equation, which gives the slope of the vaporization as a function of temperature as

$$\frac{dp}{d\Theta} = \frac{L}{\Theta \times (V_{\text{gas}} - V_{\text{liquid}})} \quad (4.1)$$

Here, the latent heat per atom is given by $L = \Theta \times (S_{\text{gas}} - S_{\text{liquid}})$, where S denotes the atomic entropy of the gas and the liquid, respectively. We have further assumed here that L does not depend on temperature which is a reasonable approximation for LHe. If we neglect the volume of the liquid (for LHe at 4.2 K, it is a factor of 750 smaller than the volume of the vapor), and model the gas as an ideal gas, $pV = nk_B\Theta$, it is found by integration of (4.1) that the vapor pressure p drops exponentially as Θ decreases, i.e.,

$$p(\Theta) = p_0 \exp\left(-\frac{L}{k_B\Theta}\right) \quad (4.2)$$

The cooling power P is simply the latent heat taken from the liquid per evaporated atom, multiplied by the number of atoms evaporated per time.

$$P = \frac{dn}{dt} L \quad (4.3)$$

Since $\frac{dn}{dt}$ is determined by the pumping speed $\frac{dV}{dT}$ of the pump used via

$$\frac{dn}{dt} = \frac{1}{m_{\text{He}}} \frac{dM}{dt} = \frac{1}{m_{\text{He}}} \rho \frac{dV}{dt} = \frac{p(\Theta)}{k_B\Theta} \frac{dV}{dt}$$

the cooling power drops exponentially as Θ decreases.

Question 4.2: The latent heat of ^4He is 88 J/mol. What is the cooling power at $\Theta = 1.2$ K when a pump with a pumping speed of 200 m^3/h is used?

The steady state is reached when the cooling power equals the heat load of the LHe. With a conventional pump with a pumping speed of, say 10 m^3/h , a temperature of about 1.2 K can be reached. Lower temperatures somewhat below 1K are possible, but require very powerful pumps. Therefore, if this temperature range is needed, people usually prefer a ^3He cryostat or a dilution refrigerator. Sometimes, temperatures *above* 4.2 K are required. The device of choice is then a *gas flow cryostat*. Here, the sample sits in a flow of cold helium gas, which enters the sample chamber via a needle valve. The sample chamber itself is thermally decoupled from the LHe by an additional vacuum chamber. The sample temperature can be adjusted by controlling the power applied to a heater for the gas, in combination with the gas flow rate. Continuous variation of the temperature between 1.2 K and room temperature is possible in gas flow cryostats. Many cryostats are equipped with a superconducting magnet, which is cooled below the critical temperature by the LHe. Most of these magnets are made from Nb alloys, since they have very large critical magnetic fields. A typical magnet is able to generate magnetic fields of the order of 10 T, although 20 T are commercially available. Experiments at higher magnetic fields can be carried out at some national and international high magnetic field laboratories.

^3He cryostats

Below 1K, the vapor pressure of ^3He is much higher than that one of ^4He . Therefore, temperatures down to about 270 mK can be reached easily by pumping L ^3He . In a ^3He cryostat, the ^3He is isolated from the ^4He precooling stage by an inner vacuum chamber (Fig. 4.26). As mentioned already, the ^3He is kept in a closed cycle. The pumped ^3He gas is collected in a storage vessel. Measurements can be performed until all the L ^3He has been pumped, which results in measurements intervals up to one day. The ^3He gas can be condensed by a small, pumped ^4He pot, which is connected to the ^4He bath via a needle valve, such that its temperature stays well below 3.2 K, the condensation temperature of ^3He . Some cryostats are equipped with a continuous flow option. Here, the pumped ^3He is immediately recondensed. For the price of a somewhat higher base temperature due to the additional heat load, the measurement period becomes unlimited this way.

$^3\text{He}/^4\text{He}$ dilution refrigerators

This type of cryostat uses the special properties of $^3\text{He}/^4\text{He}$ mixtures in a clever way, and makes possible temperatures as low as 1 mK and even below. Since the D phase of the mixture is approximately a dilute Fermi gas, it can be thought of the ^3He vapor of the C phase, with a significant vapor pressure even at $\Theta = 0$. Since the C phase has a smaller density, the "liquid" will float on top of the "gas", though. Pumping the ^3He atoms out of the D phase will surely cause ^3He from the C phase to evaporate, which pulls the corresponding effective latent heat out of the mixture. This is the cooling mechanism in a dilution refrigerator as

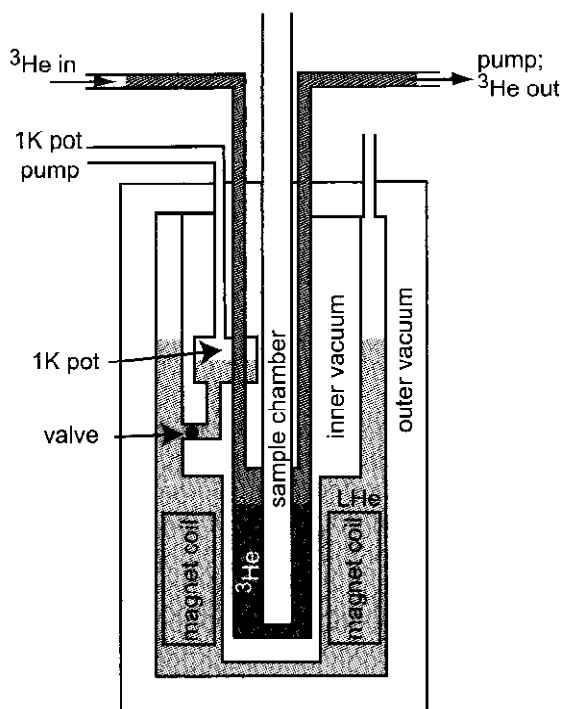


Figure 4.26: Schematic sketch of a ^3He cryostat.

sketched in Fig. 4.27. The mixture rests in the *mixing chamber*. The D phase is connected through a tube with the *still*, a pot that gets heated to about 600 mK. At this temperature, the vapor pressure of ^3He is significant, while that one of ^4He is negligible. The still therefore effectively distills ^3He from the D phase. The missing ^3He in the D phase gets delivered by “evaporation” across the C-D phase boundary, and the mixture in the mixing chamber gets colder. Usually, the evaporated ^3He is recondensed into the mixing chamber by a pot filled with ^4He , that gets pumped temperatures below the condensation temperature of ^3He . This is the “1K pot”. The freshly condensed ^3He has, of course, still a much higher temperature than the mixture. The heat flow in the mixing chamber is therefore optimized by a flow impedance in the condenser line. In addition, the outgoing gas at the still temperature is used to further precool the condensed ^3He via heat exchangers. Virtually all mesoscopic transport experiments below 270 mK have been carried out by thermally coupling the sample to the mixing chamber, either by immersing it directly, or by mounting it in the vacuum at the outside wall of the mixing chamber.