6

Helium-3 Cryostats

The temperature range accessible with a liquid ^{4}He bath is typically 4.2–1.3 K, but this temperature range can be extended to about 0.3 K if the rare isotope ^{3}He is used instead of the common isotope ^{4}He. The main reason is that ^{3}He has a substantially larger vapour pressure than ^{4}He at the same temperature; the ratio P_3/P_4 is 74 at 1 K but about 10^4 at 0.5 K (Figs. 2.7 and 2.8). A further advantage of using liquid ^{3}He instead of liquid ^{4}He at temperatures below their normal boiling point is due to the fact that the specific heat of liquid ^{3}He varies much less between, for example, 2 and 0.5 K than the specific heat of liquid ^{4}He does (Fig. 2.9). One therefore has to evaporate only about 20% of ^{3}He to cool this liquid from 1.5 to 0.3 K by using its own heat of evaporation. Furthermore, the specific heat of liquid ^{3}He is larger than the specific heat of liquid ^{4}He below 1.5 K, resulting in a larger heat reservoir in this temperature range. Finally, liquid ^{3}He is not superfluid in the temperature range of concern in this chapter. One therefore does not have the heat transfer problems sometimes arising from the superfluid film flow of liquid ^{4}He (Sect. 2.3.5).

There are two rather serious disadvantages of liquid ^{3}He. Firstly, its latent heat of evaporation is substantially smaller than that of liquid ^{4}He, see Fig. 2.6. Secondly, and more importantly, ^{3}He is much more expensive than ^{4}He. A typical price is about 200 € per liter gas or about 150 € per cm^3 liquid (a liter of liquid ^{4}He, if bought commercially, can be obtained for about 6 €). Due to this high price, which results from the expensive production method of ^{3}He (Sect. 2.3.1), one can only use ^{3}He in a closed gas handling and cryogenic system, making sure that no gas is lost. As a second consequence, one never has enough ^{3}He gas to liquefy it on a technological scale in a liquefaction plant. The ^{3}He is transformed from the gaseous to the liquid state in the cryostat in which it will be used for doing experiments just by bringing it in contact with a ^{4}He bath at a temperature of, say, 1.3 K. This temperature can be obtained by the continuously evaporating ^{4}He refrigerator discussed in Sect. 5.2.4. As the critical temperature of ^{3}He is 3.3 K, the ^{3}He gas will condense on surfaces which are below this temperature if the gas pressure is high enough.

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Because the use of liquid ^{3}He as a refrigerant occurs in a ^{4}He cryostat anyway, all the parts refrigerated by the ^{3}He can be surrounded by shields kept at ^{4}He temperature. In addition, all the tubing and wiring going to the experiment can be thermally heat sunk at the ^{4}He bath. Therefore, the heat transferred from the outside world to parts below 1 K can be absorbed by the ^{4}He bath, which has a substantially larger volume and also a larger heat of evaporation than the ^{3}He bath. The ^{3}He bath is then only used for cooling from the temperature of the ^{4}He bath to the temperature obtained by the ^{3}He refrigerator. In any case, the use of an evaporating ^{3}He bath is the simplest way of reaching temperatures between 0.3 and 1 K, and several suppliers offer ^{3}He cryostats commercially. Reports on ^{3}He cryostats can be found in [6.1–6.16].

6.1 Helium-3 Cryostats with External Pumps

Figure 6.1 schematically presents typical designs of ^{3}He cryostats in the order of increasing sophistication. In each design a few cm^3 of ^{3}He are liquefied by bringing the ^{3}He in thermal contact with a ^{4}He bath, which is pumped to $T \leq 1.5\,\mathrm{K}$. Figure 6.1a shows a setup where the pre-cooling stage is the main ^{4}He bath pumped to a temperature of about 1.3 K and which absorbs the latent heat of condensation of the incoming ^{3}He. The ^{3}He gas condenses on the cold surfaces of the thin-walled pumping tube leading to and supporting the ^{3}He pot (surrounded by vacuum), which will slowly cool and eventually collect liquid ^{3}He. When all the ^{3}He has condensed we pump on this liquid to reduce its temperature from about 1.3 K to the desired temperature; the minimum is typically 0.3 K.

In the second setup the main ^{4}He bath is not pumped but is left at normal pressure and 4.2 K, and to condense the ^{3}He we use a continuously evaporating ^{4}He refrigerator, as discussed in Sect. 5.2.4. Both designs utilize the single-cylce discontinuous refrigeration method for the ^{3}He part because eventually all the ^{3}He is evaporated (and is hopefully recovered!) and we have to recondense it to start again.

Finally, in the third design the ^{3}He refrigerator, too, is run in a continuous mode by introducing a recondensing tube. The ^{3}He vapour that we pump away from the liquid ^{3}He bath at its vapour pressure will leave the room-temperature pump at a pressure of several 0.1 bar, it is pre-cooled by the ^{4}He pot and eventually recondensed into the ^{3}He pot. For this purpose the ^{3}He gas will run through a heat exchanger in the 4.2 K bath as well as a second heat exchanger in the 1.3 K ^{4}He bath where it will, of course, condense. The now liquid ^{3}He will then be isenthalpically (not isothermally) expanded through an impedance of order 10^{12}–$10^{13}\,\mathrm{cm}^{-3}$, which maintains a pressure sufficient for condensation before the ^{3}He arrives as a low-pressure, low-temperature liquid at the pumped ^{3}He pot. In such a design we have introduced several features (like the heat exchanger and impedances) which

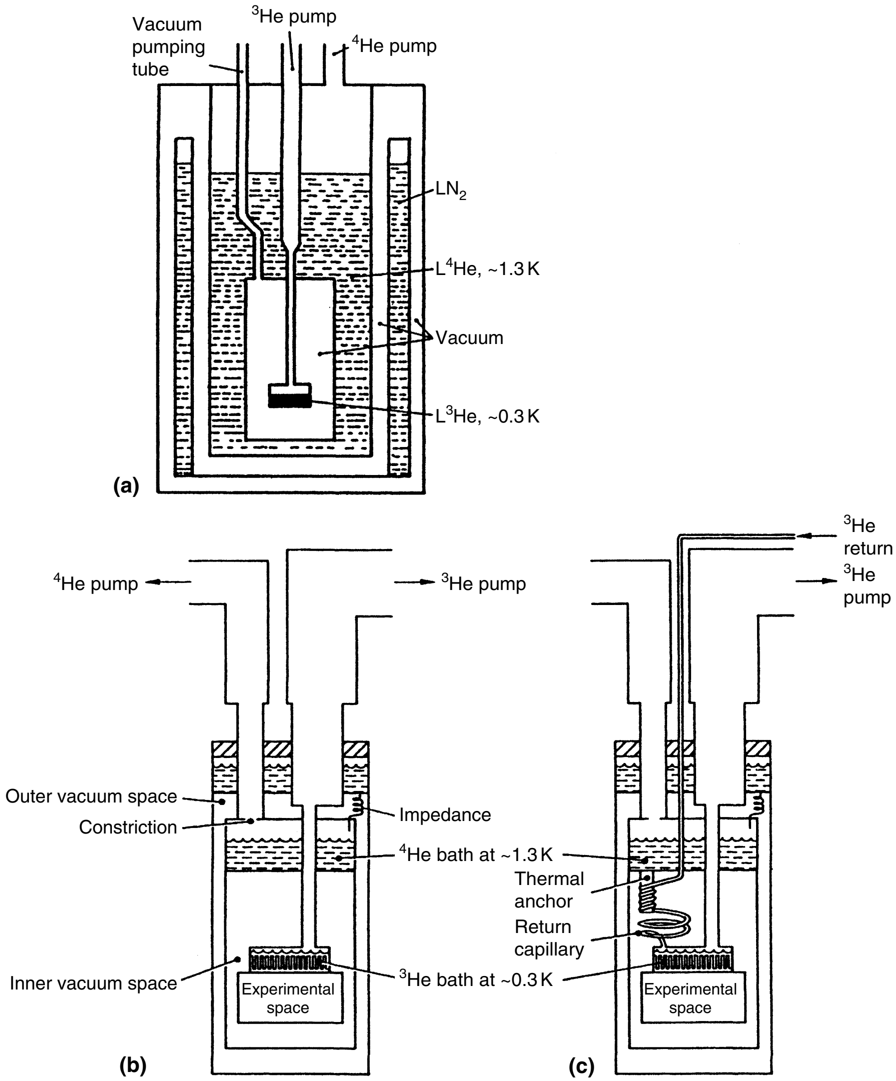

Fig. 6.1. ^{3}He cryostats of increasing sophistication. (**a**) Non-recirculating ^{3}He refrigerator with a pumped main ^{4}He bath. (**b**) Non-recirculating ^{3}He refrigerator with a continuously operating ^{4}He evaporator. (**c**) Recirculating ^{3}He refrigerator with a continuously operating ^{4}He evaporator [6.4]

will be discussed in more detail in Chap. 7 on ^{3}He–^{4}He dilution refrigerators. The narrow capillaries used as impedances can easily be blocked if there are impurities such as frozen air in the ^{4}He or ^{3}He entering them. One therefore has to be careful to avoid these impurities and, in particular, one may use a LN$_2$ cooled trap (Fig. 7.25) after the ^{3}He room-temperature pump to freeze out oil vapour or oil crack products possibly leaving the pump together with the ^{3}He gas, as well as any remaining air impurities. It is quite obvious that due to the high price of ^{3}He gas it is necessary to have a vacuum-tight closed ^{3}He system with a sealed pump avoiding any loss of this expensive gas. In addition, one has to be careful to design the room-temperature part with the smallest possible volumes, so that not too much of the ^{3}He gas remains unused in these "dead" volumes.

Due to the low helium-vapour pressure in the sub-Kelvin temperature range, one has to use reasonably dimensioned pumps as well as pumping tubes to circulate the required amount of ^{3}He. Let us consider what we need to maintain temperatures of 0.5 or 0.3 K. At these temperatures the vapour pressure of ^{3}He is about 0.2 mbar and 2 µbar, respectively. The former pressure can be maintained by a mechanical pump, whereas for the latter one we need a combination of an oil-diffusion pump and a mechanical pump. We will see that very often the limitation is not the pump but rather the conductance of the pumping tubes. Let us assume that we need a cooling power of $\dot{Q} = 1\,\mathrm{mW}$. Taking the latent heat of evaporation of ^{3}He (see Fig. 2.6), this requires an evaporation rate of

$$\dot{V} = \dot{Q}/L \simeq 5\,\mathrm{cm}^3\,\mathrm{liq}\ {}^3\mathrm{He}\,\mathrm{h}^{-1} \simeq 3\,\ell\,\mathrm{gas}\ {}^3\mathrm{He}\,\mathrm{h}^{-1}\ \text{at}\ P = 1\,\mathrm{bar}\,. \tag{6.1}$$

At the pressures mentioned above, the volume flow rates have to be 15 and $1,500\,\mathrm{m}^3\,\mathrm{gas}\,\mathrm{h}^{-1}$, respectively. These volume rates are no problem for a mechanical pump and a diffusion pump, respectively. But what about the tubing? The conductance $L[\mathrm{m}^3\,\mathrm{h}^{-1}]$ of a tube for a laminar flow is given by

$$L = 486\bar{P}d^4/l\,, \tag{6.2}$$

where d is the diameter [cm], l is the length [cm], and $\bar{P}$ is the mean pressure [mbar]. We then find that we need pumping tubes with diameters of 3 and 10 cm, respectively, if the length is several meters. A pumping tube with the latter diameter is bulky and one should rather try to reduce the heat input to the ^{3}He system to below 1 mW if temperatures below 0.5 K are required. Inside the cryostat, the diameter of the pumping tube can be reduced according to the temperature profile because the density of the evaporating gas increases with decreasing T and the circulation rate $\dot{n}$ is the same everywhere [6.4,6.14].

6.2 Helium-3 Cryostats with Internal Adsorption Pumps

One can avoid the room-temperature pump as well as the often-bulky pumping tubes by inserting a cold adsorption pump inside the cryostat. Gases adsorb at cold surfaces if their temperature is low enough. If we keep a large surface at a low enough temperature above our ^{3}He bath, this surface will pump the helium vapour and keep the liquid ^{3}He at a low temperature [6.6–6.16]. There are various suitable materials (e.g., charcoal, zeolites or fine metal powder) with surface areas of at least several $m^2\,g^{-1}$ [6.10,6.11,6.17] (see also Sect. 13.6). If we fill a volume of several cm^3 with such an adsorbent with large surface area at low temperature, it will very effectively pump the liquid ^{3}He bath. When all the ^{3}He has been pumped away, so that the ^{3}He pot is empty, we just have to lift the charcoal pumping system into a space at higher temperature in the cryostat to desorb the helium, which will then enter the gas phase, condense at the cold surfaces of the cryostat and eventually drip back down into the ^{3}He pot. Such a cryostat with a hermetically sealed ^{3}He system reaching 0.25 K is shown in Fig. 6.2. An alternative way of switching between the pumping and the releasing state of the adsorbent (instead of lifting and lowering this part) is to switch the thermal contact of the adsorbent with the surrounding ^{4}He bath on and off. This can be done by using helium exchange gas in the

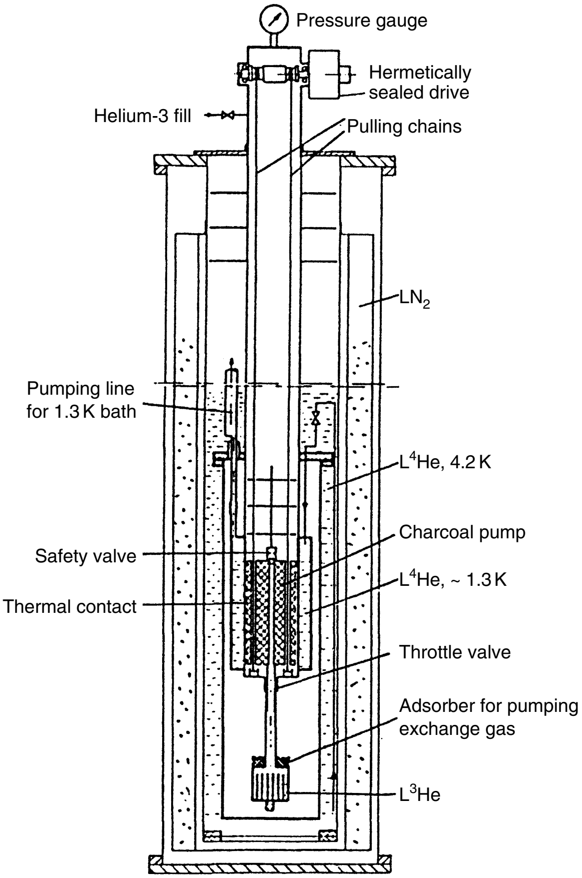

Fig. 6.2. Hermetically sealed, charcoal pumped ^{3}He refrigerator. The charcoal pump can be raised or lowered by means of a chain drive operated via a hermetically closed rotating seal [6.11]

space around the vessel containing the adsorbent and pumping this exchange gas away when the adsorbent has to be thermally isolated and heating it by a heater to a higher temperature to release the ^{3}He gas. We have to bear in mind that the heat of adsorption is rather large – of the order of the latent heat of evaporation – so the adsorbent has to be in good thermal contact with the ^{4}He bath at about 1.4 K to remove the heat of adsorption and avoid an unwanted high temperature of the adsorbent. The pumping speed of charcoal pumps is not only a function of its temperature but also, in practice, a complicated function of geometry, thermal coupling and pressure [6.10, 6.11, 6.17, 6.18].

Cold charcoal pumping systems are also more efficient than room-temperature mechanical pumps because they are connected via a short, cold pumping tube to the ^{3}He pot, taking advantage of the very high pumping speed of the adsorbent (see below).

A very versatile ^{3}He cryostat which can be inserted into a storage ^{4}He dewar (50 mm neck diameter) has been designed by Swartz [6.14, 6.15], as well as in our laboratory at the University Bayreuth (Fig. 6.3). It is an extension of the "^{4}He dipper cryostat" mentioned in Sect. 5.2.4 (Fig. 5.6). The first stage of the cryostat is a continuously filling ^{4}He pot at about 1.3 K, on which the ^{3}He condenses out of its small room-temperature storage volume on top of the cryostat and then drips into the ^{3}He pot. Cooling of the ^{3}He to 0.3 K is achieved (within a few minutes!) by pumping with an activated charcoal pump which is located inside the cryostat close to the ^{3}He pot. The charcoal pump is equipped with its own (charcoal pumped) vacuum/exchange gas space and heater so that its temperature can be regulated between about 5 and 25 K to adsorb or desorb the ^{3}He. The small dead volumes allow the quantity of ^{3}He to be restricted to just 1 STP liter of ^{3}He (about 1.5 cm^3 of liquid) in the permanently sealed ^{3}He part of the cryostat. The pump and heaters can be computer controlled. The hold-time of the ^{3}He charge in the pot depends, of course, on the heat load; typical times are from 3 h for $\dot{Q} \approx 0.1$ mW to about 20 h with no external load. Such a portable ^{3}He cryostat requires no transfer of liquid ^{4}He and no external gas handling or pumping system for the ^{3}He part, it has a very fast turn-around time and low cost. The design and construction, as well as the operation and performance, are described in detail in [6.15]; it is available commercially. The novel design of a compact adsorption pumped ^{3}He cryostat with a minimum temperature of 0.24 K, a hold time of 30 h at a heat load of 0.1 mW, and a cooling power of 1 mW at 0.3 K described in [6.16] is also commercially available.

The cooling power of 20 g of activated charcoal as a function of the heating power is shown in Fig. 6.4, while Fig. 6.5 displays the "pumping power" of activated charcoal. Figure 6.4 shows that one can maintain temperatures as low as 0.25 K for heating rates below about 0.01 mW, and 0.4 K at 1 mW.

Of course, cold materials with a large surface area are not only useful as a pump for reducing the temperature above an evaporating cryogenic liquid. They are also quite useful in the vacuum space of cryogenic vessels, where they can substantially improve the vacuum by adsorbing remaining gas particles

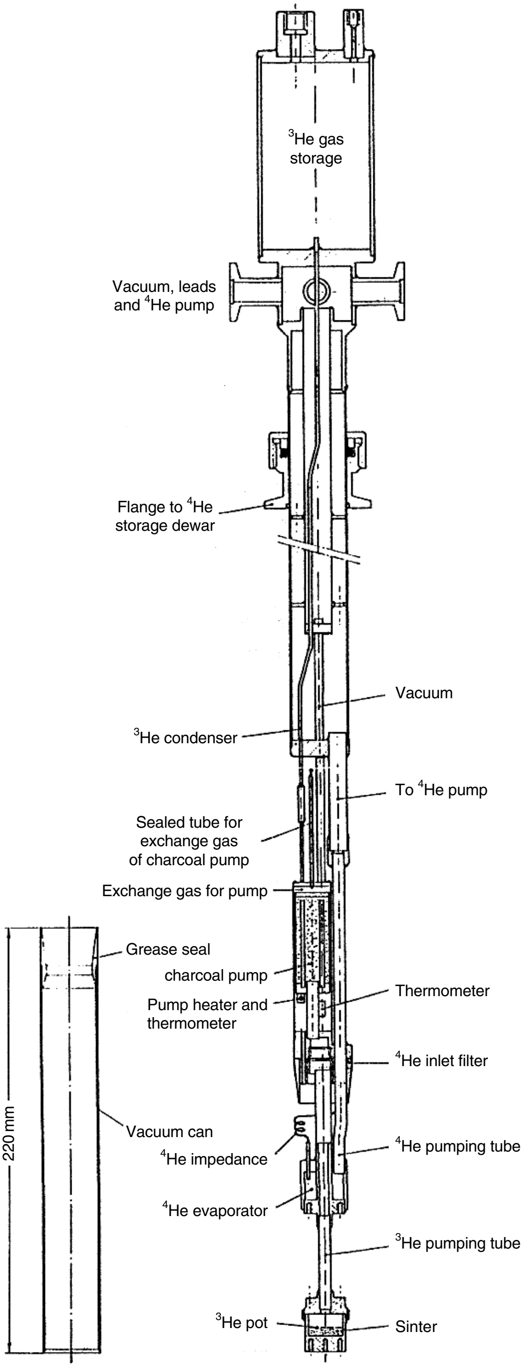

Fig. 6.3. ^{3}He dipstick refrigerator with ^{3}He gas storage, charcoal pump, continuously operating ^{4}He refrigerator and ^{3}He refrigerator; for details see text and [6.15] (courtesy of P. Sekowski, Universität Bayreuth)

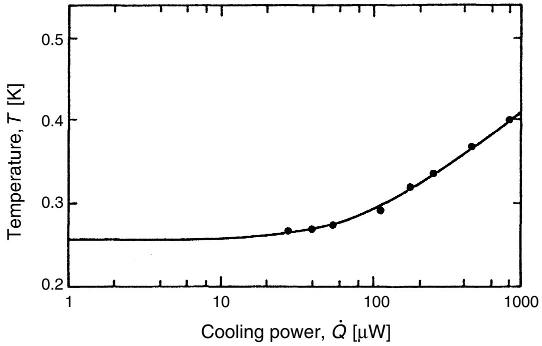

Fig. 6.4. Cooling power of a ^{3}He refrigerator pumped by 20 g of charcoal [6.11]

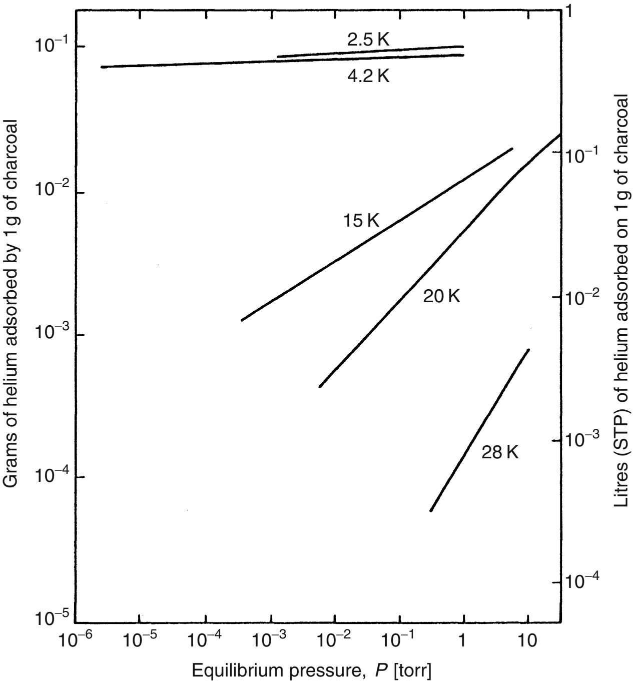

Fig. 6.5. Adsorption isotherms of ^{3}He on activated charcoal as a function of the helium-gas pressure [6.11]

after cooling to low temperatures, or even make the gas entering the vacuum space through a tiny, not localized leak "harmless".

With a ^{3}He cryostat we are taking the first step into the range of $T < 1\,\mathrm{K}$. At these temperatures the Kapitza thermal boundary resistance (Sect. 4.3.2) can be of importance. One should, therefore, increase the surface area of the ^{3}He pot to improve the thermal coupling between the cryoliquid and the wall of its container. This can be done by making grooves in the inside of the ^{3}He pot or, better, by sintering fine metal powder (Sect. 13.6) with a total surface area of about 1 m^2 to the bottom of the container.

Helium-3 cryostats were quite popular until about the end of the 1960s, when the ^{3}He–^{4}He dilution refrigerator (see Chap. 7) was invented. This type of refrigerator reaches substantially lower temperatures. Today, ^{3}He refrigerators are mainly used if only a simple setup is possible or necessary, or when one needs a very high cooling power at temperatures between about 0.4 and 1 K.

Problems

6.1. Calculate how much of a liquid ^{3}He bath has to be evaporated to cool it from 1 to 0.3 K.

6.2. How much liquid ^{3}He has to be evaporated to refrigerate 1 kg of Cu from 1 to 0.3 K?

6.3. Calculate the cooling power of a ^{3}He refrigerator at $T = 0.3\,\mathrm{K}$, if a $100\,l\,\mathrm{s}^{-1}$ pump is used (see Fig. 2.6 for the latent heat of evaporation and Fig. 2.7 for the vapour pressure).

6.4. Calculate the surface area of a sintered Ag heat exchanger necessary for a ^{3}He cryostat operating at 0.3 K and $\dot{Q} = 0.1\,\mathrm{mW}$ for the data displayed in Fig. 4.6 and extrapolated from Fig. 4.7.

6.5. How many grams of charcoal does one need to keep a ^{3}He refrigerator running at a heat input of 0.2 mW at 0.5 K?