Approach to Absolute Zero

2. From 4.22 K to 0.3 K

R Srinivasan

R Srinivasan is a Visiting Professor at the Raman Research Institute after retiring as Director of the Inter-University Consortium for DAE Facilities at Indore. Earlier, he was at the Physics Department of IIT, Chennai for many years. He has worked in low temperature physics and superconductivity.

The previous article of this series was:
1. Liquefaction of gases, December 1996.

The liquefaction of gases was described in Part I of the series. The normal boiling point of ^4He is 4.22 K. By boiling a cryogenic liquid under low pressure one can attain a temperature lower than the normal boiling point. Since the vapour pressure of a liquid cryogen decreases rapidly with decreasing temperature, a limit is set to the lowest attainable temperature by pumping on a cryogenic bath. With a liquid ^4He bath this is 1.2 K. However using a bath of liquid ^3He one can attain a temperature as low as 0.3 K.

Introduction

The liquefaction of gases to attain low temperatures was dealt with in Part I of this series of articles. Liquid helium has a low boiling point of 4.22 K under a pressure of one atmosphere. As the second step in reaching still lower temperatures, one resorts to pumping on a liquid helium bath. In this part of the series such methods to achieve temperatures down to 0.3 K will be described.

Although the principle involved sounds simple, practical implementation requires meticulous attention to many details. If proper attention is not paid to any one of the details the required low temperature will not be attained. The rest of the article is intended to give some feel for the challenge of experimental low temperature research.

Pumping on a Liquid Cryogen Bath

One can reduce the pressure on a liquid by pumping on it with a vacuum pump. This causes the boiling point of the liquid to come down. At the boiling point the enthalpy of the vapour is more than the enthalpy of the liquid by an amount equal to the latent heat of vaporisation of the liquid. When the liquid vaporises, it absorbs heat from the bath and produces cooling. If n is the number of moles of the liquid evaporated per second and L the latent heat of vaporisation in Joules per mole, the cooling produced in watts is given by

$$Q = n L$$

If the pumping speed of the pump is S and the density of the vapour is σ, then n is proportional to $S\,\sigma$. The density σ of the vapour is proportional to its vapour pressure p. As the temperature falls the vapour pressure decreases rapidly (*Figure 1*). To reach still lower temperatures one should use a pump with an even larger pumping speed. The effective pumping speed on the bath not only depends on the speed of the pump but also on the length and the diameter of the connecting tubes. In a cryostat there is a minimum length for the pumping path and so, as the temperature is lowered, one has to use tubes of larger and larger diameter for connection. This sets the limit on the lowest

Pumping on a cryogenic liquid bath lowers its temperature.

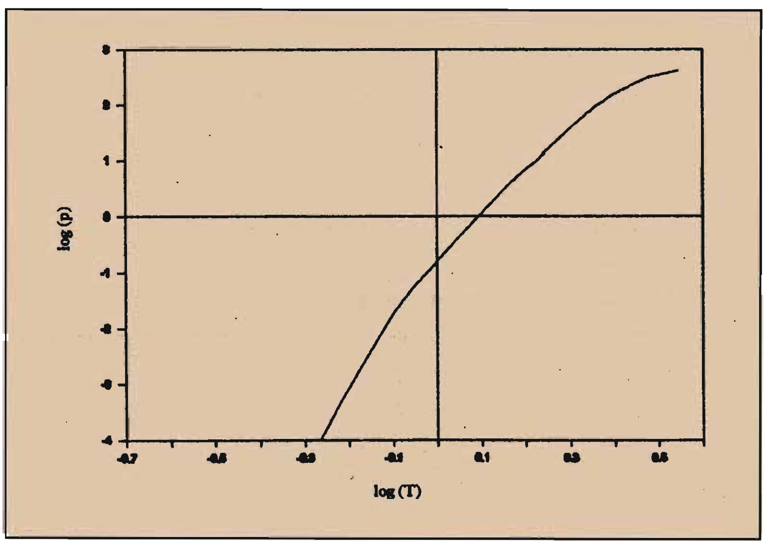


***Figure 1** Temperature dependence of the vapour pressure of liquid ^{4}He.*

temperature which one can reach with tubes of reasonable diameter for pumping on the cryogenic liquid bath. With liquid helium the lowest temperature one can reach is around 1.2 K.

When the temperature of liquid helium falls below 2.17 K it becomes a superfluid.

When the temperature on the liquid helium bath falls below 2.17K, a dramatic change in the properties of liquid helium is observed. This temperature is called the *Lambda transition temperature* because the graph of the temperature variation of specific heat of the liquid around this temperature resembles the Greek letter Lambda. Above this temperature one sees vigorous boiling of the liquid with vapour bubbles rising continuously to the surface of the liquid. The vapour bubbles are nucleated due to temperature fluctuations in the liquid. When the temperature of the liquid drops below 2.17K no bubbles are seen in the liquid and the surface appears very quiescent. The thermal conductivity of liquid helium II (liquid helium below the Lambda point) is a million times larger than the thermal conductivity of liquid helium I (liquid helium above the Lambda point). Temperature fluctuations cannot arise in liquid helium II and so no bubbles are nucleated. Liquid helium II shows no viscous resistance to flow through narrow channels. This is illustrated in a dramatic fashion by the fountain effect experiment described in the later box.

A Typical Low Temperature Experimental Set-up

A typical low temperature experimental set-up to measure some physical property, say the electrical resistivity, of a sample down to 1.8 K is shown in *Figure 2*. The sample (6) is mounted at the end of a long thin-walled stainless steel (SS) tube (19) which is suspended from a flange at the top of the cryostat through a Wilson seal (20). The Wilson seal will allow the up and down motion of the SS tube in vacuum. The sample is surrounded by a vacuum can (5). The sample space can be evacuated, if necessary, by a vacuum pump connected to the port (16). This enables measurements to be done from the temperature of the liquid helium bath up to room temperature. This port (16) can also be

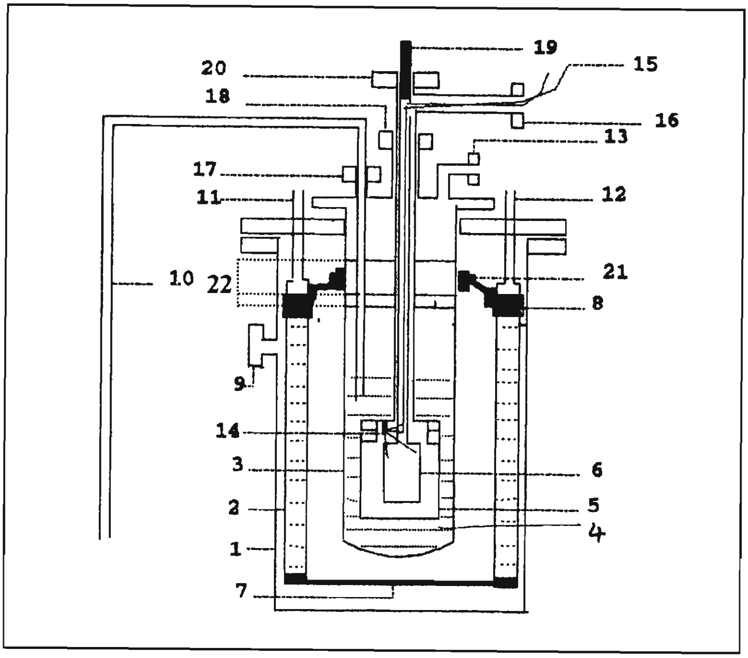


Figure 2 A typical cryostat for measurement of electrical resistivity in the range 1.8 to 300 K.

used to bring out the electrical leads (15) to the thermometer and the sample. The vacuum can is suspended in a bath of liquid helium (4) in a SS dewar (3) which is again suspended from the top flange of a vacuum chamber (1). In order to keep the evaporation rate of liquid helium low it is necessary to reduce the heat leaking to the helium bath from the surroundings at room temperature. Heat can be transferred by convection, conduction and radiation. By evacuating the vacuum chamber (1) to a pressure of 10^{-6} millibar by connecting a vacuum pump at the port (9) convective heat transfer to the liquid helium is reduced. At such low pressures the mean free path of the gas molecules is larger than the distance between the walls. Under such conditions heat conduction by the gas molecules becomes proportional to pressure and is negligible compared to heat conduction through the material of the cryostat or by radiation. The helium vessel as well as the supporting tube of the sample chamber is made of thin walled (wall thickness approximately 0.2 mm) stainless steel tubing. Stainless steel has a low thermal conductivity and good mechanical strength. By making the wall thickness small the area of cross-section for heat conduction is reduced. The heat radiation reaching an object at temperature T_2 from the surroundings at temperature T_1 is proportional to $(T_1^4 - T_2^4)$. The helium dewar is surrounded by an annular vessel (2) in which liquid nitrogen is filled through the tube (11). Tube (12) acts as the vent for the evaporating nitrogen gas. A copper plate (7) in good thermal contact with the liquid nitrogen bath covers the bottom of the helium vessel. The liquid nitrogen vessel at 77K and the cold copper plate surrounding the helium bath reduce the heat radiation reaching the helium bath. The radiation from the room temperature wall passes into the liquid nitrogen bath and evaporates liquid nitrogen. Since liquid nitrogen is cheaper than liquid helium one would prefer that liquid nitrogen be consumed rather than liquid helium. If the liquid nitrogen shield is absent heat radiation from the wall of the vacuum chamber (1) at room temperature (T_1, approximately 300 K) will reach the liquid helium bath. When the helium bath is surrounded by liquid nitrogen (temperature of the liquid nitrogen bath is 77 K) the heat radiation reaching the helium bath is reduced considerably. A braided copper wire (21) soldered at one end to the copper bush (8) on the liquid nitrogen vessel is wrapped on the outside of the neck of the SS vessel containing helium. In addition copper baffles (22) are soldered to the SS tube supporting the vacuum can (5). These arrangements serve to reduce the heat radiation travelling down the helium vessel. A liquid helium transfer tube (10) passing through the Wilson seal (17) is used to transfer liquid helium from the storage dewar to the cryostat. A recovery system connected to the port (13) serves to collect the evaporating helium gas. A vacuum pump connected to the same port suitably can be used to reduce the pressure on the helium bath and to lower its temperature. All electrical leads to the thermometer and the sample need to be properly anchored to a point at 4.2 K. These wires are wrapped around a copper stud (14) on the top flange of the vacuum can (5) as shown in the figure. All the heat conducted from the room temperature end by the electrical leads will pass into the liquid helium bath. In any low temperature experiment care must be taken to reduce the heat leaking from high temperatures to the cold sample. The precautions have to be more stringently observed as one attains

In any low temperature experiment care must be taken to reduce the heat leaking from high temperatures to the cold sample. The precautions have to be more stringently observed as one attains lower temperatures.

lower temperatures.

The above detailed description indicates that any low temperature experiment involves careful design of the cryostat and requires good experimental skill.

Liquid ^3He

Helium has two isotopes. ^4He, with mass number 4, is the more abundant isotope. Its nucleus has two protons and two neutrons and the nuclear spin is zero. Therefore it obeys Bose-Einstein statistics. Whenever we referred in the above paragraphs to liquid helium it was tacitly understood that we were referring to liquid ^4He. The superfluid property exhibited by liquid ^4He is a consequence of Bose condensation exhibited by particles obeying Bose-Einstein statistics. The other isotope is ^3He. This isotope is present in about one part per million in natural helium. It has two protons and one neutron in the nucleus. Its spin is 1/2. It is a Fermion. This isotope is artificially produced in nuclear reactors used in the manufacture of tritium, the isotope ^3H of hydrogen. The cost of ^3He gas is about 15,000 to 20,000 times the cost of natural helium. The normal boiling point of ^3He is 3.19 K and its critical temperature is 3.32 K. So ^3He gas under pressure, and cooled to 1.2 K in a pumped liquid ^4He bath, will liquefy. The

Liquid ^3He has a higher vapour pressure than liquid ^4He. By pumping on a liquid ^3He bath one can obtain a temperature as low as 0.3 K.

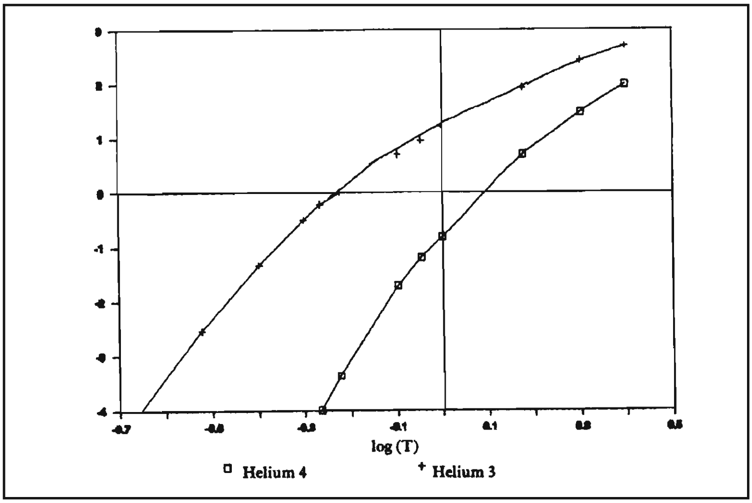


Figure 3 Vapour pressure curves of liquid ^3He and liquid ^4He.

vapour pressure of liquid ^3He is substantially higher than the vapour pressure of ^4He at any given temperature (*Figure 3*). With a vacuum pump of a given pumping speed, there will be a larger throughput of ^3He compared to that of ^4He below 1 K. So it will be possible to reach a lower temperature with a pumped liquid ^3He bath than with a pumped ^4He bath. One can reach a temperature as low as 0.3 K by pumping on a liquid ^3He bath. Below this temperature the vapour pressure is too low for effective pumping with reasonable capacity pumps and connecting tubes of reasonable diameter

A schematic diagram of a ^3He cryostat is shown in *Figure 4*. This is a continuous refrigerator in which the evaporating ^3He vapour that is pumped by a vacuum pump is recondensed in the ^3He pot. The ^3He gas at a few hundred millibars pressure at room temperature is first cooled in a heat exchanger immersed in a ^4He bath at 4.2 K. The cold ^3He gas then passes through a ^4He bath boiling under reduced pressure at 1.3K. It is then is enthalpically expanded (J-T expansion) through a narrow capillary which offers an impedance of 10^{12} to 10^{13} cm^{-3} to the flow. In the capillary the pressure is sufficiently high for ^3He to liquefy. The flow impedance of the capillary is large enough to sustain the pressure differential necessary so that the liquid ^3He in the pot is boiling at 0.3 K under reduced pressure due to suction by the vacuum pump. The sample is attached to the ^3He pot and the measurements are done on the sample.

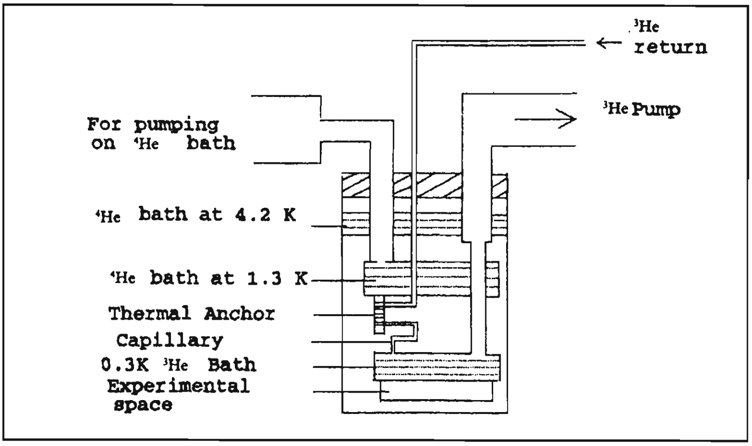
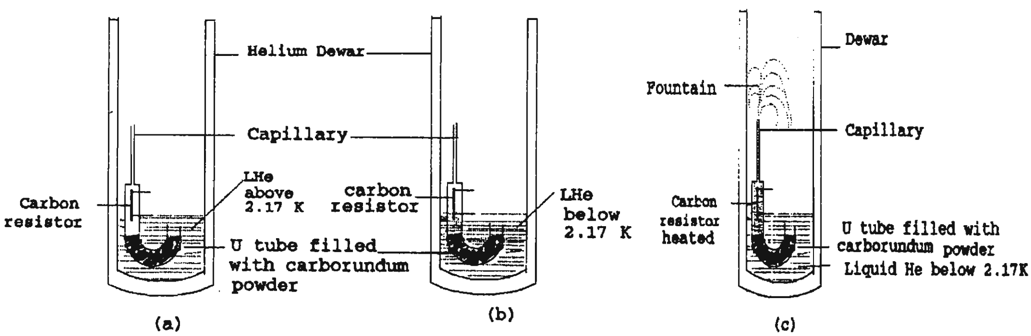


Figure 4 A schematic diagram of a continuously operating liquid ^3He cryostat to reach down to 0.3 K.

Fountain Effect in Liquid ^{4}Helium

When the temperature of liquid helium falls below 2.17 K it behaves as a *superfluid*. This is demonstrated in a dramatic fashion by the fountain effect experiment. A U shaped tube has a capillary attached to it at one end as shown in *Figure* (i). The wider part of the U tube is filled with fine powder of carborundum which is closely packed. Below the capillary tube a small carbon resistor is fixed. When the tube is suspended in a liquid helium bath above 2.17 K, with the wider end below the liquid helium level and the capillary projecting above it, no liquid enters the tube (*Figure*.i(a)). The narrow pores in the tightly packed tube offer a very large viscous resistance to the flow of liquid helium above 2.17 K. The hydrostatic pressure is insufficient to overcome the flow resistance. However when the liquid helium is cooled below 2.17 K by pumping on the bath, one observes liquid entering the tube and filling both sides of the U tube to the same level (*Figure*.i(b)). This is because part of the liquid helium has become a superfluid which can flow through capillaries without any flow resistance. At any temperature both normal and superfluid components co-exist in equilibrium. The proportion of the normal component decreases and the proportion of the superfluid component increases as the temperature falls below the Lambda point. If now a small current is passed through the carbon resistor to generate a few milliwatts of heat, liquid helium comes out of the capillary as a fountain (*Figure*.i(c)). The larger the heat supplied to the resistor the more the height of the fountain. When the resistor is heated, the concentration of the superfluid component in the immediate vicinity of the resistor falls below that in the bath. Superfluid helium rushes into the region near the resistor. However the normal fluid near the resistor cannot flow out because of the viscous resistance. So a pressure builds up leading to the ejection of the liquid as a fountain through the capillary.

Figure (i)



Since ^3He gas is very costly one will use only a few cc and the entire system must be absolutely leak tight to avoid any loss.

Since ^3He gas is very costly one will use only a few cc and the entire system must be absolutely leak tight to avoid any loss. As one uses a very narrow capillary to generate the large flow impedance, the ^3He gas should not be contaminated with traces of impurities which will freeze and block the capillary.

Commercial ^3He inserts in ^4He cryostats are available to carry out measurements of transport properties down to 0.3K in applied magnetic fields. Commercial ^3He cryostats are available in the Indian Association for Cultivation of Science in Calcutta and the Indian Institute of Technology, Chennai. In the Department of Physics, Indian Institute of Science, Bangalore, a home-built ^3He cryostat has been used in measurement of transport properties.

In the next two parts of this series we will describe techniques to reach still lower temperatures. It is the development of such techniques that made the discovery of superfluidity in ^3He possible. The Nobel Prize in Physics for 1996 was awarded to Lee, Osheroff and Richardson for this discovery.

Address for Correspondence
R Srinivasan
Visiting Professor
Raman Research Institute
C V Raman Avenue
Sadashivanagar
Bangalore 560 080, India
rsv@rri.ernet.in

Suggested Reading

- G K White. *Experimental techniques in low temperature physics*. OUP, 1959.
- A C Rose-Innes. *Low temperature techniques*. English University Press, 1973.
- Frank Pobell. *Matter and methods at low temperatures*. Springer Verlag, 1992.
- R Srinivasan.The Nobel Prize in Physics - 1996. *Resonance*. Vol.1.12. 65, 1996.

Every great advance in science has issued from a new audacity of imagination.

John Dewey