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In this paper the authors describe a novel approach to the use of the cryopump in obtaining temperatures in the region below 1° K and indicate some of the advantages.

HELIUM-3 CRYOSTAT WITH ADSORPTION PUMP

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MECHANICAL or mercury diffusion pumps are generally used in conjunction with helium-3 refrigerators. Cryopumps have been used to a minor extent; their advantages are a considerable reduction of vibrations which generate heat, and a tight circuit.[1–4] In contrast to the usual cryopump design we completely separated the adsorption device (pump) kept at 4·2° K, from the cooling device (cryostat) at 1·2° K. This avoids an additional thermal load for the helium-4 bath. It also facilitates the repetition of cooling cycles.

The Cryostat

Figure 1 is a diagram of the cryostat. The solid copper container 1 for helium-3 has a cavity of 3 cm³ volume to which is welded a copper-foil 2 for lowering the thermal resistance between the liquid helium-3 and the container block. A stainless steel capillary 3 measures the pressure over the helium-3 bath. Two pumping tubes 4, only one being shown, provide rigid suspension for the block and issue from the calorimetric enclosure by two copper sections connected to a radiation trap 5 with sloping baffle plates for draining the helium-3 condensed by contact with the bath at 1·2° K.

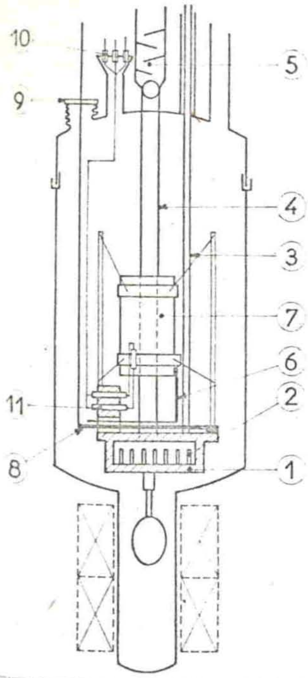

Figure 1. Helium-3 cryostat

All the pumping lines consist of tube sections of increasing diameter up to room temperature, thus reducing the admission of heat to the bath without lowering the pumping rate.

For specific heat measurements a copper tongue 6 connected to the sample 7 is pressed against the block by a gilt copper blade 8 actuated from outside by a stainless steel wire that passes through a tombac bellows 9. In order to reduce the intake of heat by the sample, the measuring wires from the helium-4 pass into the calorimeter through glass–metal plugs 10 and are welded to varnished copper wires 11 (1·5 mm diameter) fixed in holes on the helium-3 block.

Adsorption Studies

Adsorption has been studied in the field of physicochemical research[5,6] and in the field of applications, such as those described here.[7] Tests by Stern et al. with helium-4 have shown that of all the adsorbents mentioned the Linde zeolite 5A is the most suitable (saturation and pumping rate). After having ascertained the characteristics for helium-3 we adopted this adsorbent.

The adsorption cell used for pumping consists of a stainless steel pipe, 13–14 mm diameter, with a coaxial cylinder made of copper gauze, of 7 mm diameter. The zeolite 5A is in the annular space, as cylindrical granules previously degassed in a vacuum for 24 h by means of a heater coil wound around the pipe. The whole channel can be plunged into a vessel containing a cryogenic fluid (hydrogen or helium). Facilities for pumping these 2 baths to lower the temperature are provided.

The circuit comprises two tanks V_1 and V_2, holding 5·5 and 13·5 l., respectively, of stainless steel and containing

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0·5 l. helium-3 (at s.t.p.), and connected to a mercury gauge. The V_0 volume to which they are connected comprises two pressure measurement devices: a McLeod gauge fitted with liquid nitrogen trap and a Médiovac gauge (Pirani type) calibrated with the other gauge for continuous measurements at constant volume. The adsorption pump is connected to V_0 by a short large bore mains. The circuit is connected to a secondary pump, and a valve serves for recharging with helium-3.

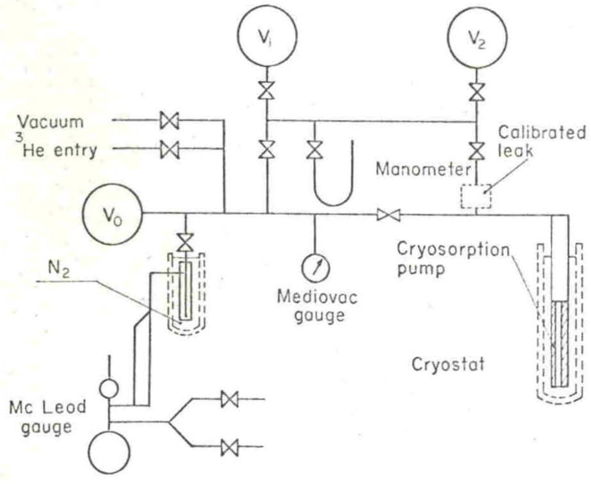

Figure 2. Apparatus for adsorption equilibrium and pumping speed measurements

The adsorption isotherms were plotted by a very well known volumetric method. Successive quantities of gas from V_1 or V_2 were admitted into V_0, of known volume, whereupon V_0 was connected to the cryopump. Measurement of the initial and final pressures in this volume gives the quantity of gas contained in the cryopump at equilibrium pressure. From this must be deducted the effective clearance volume of the cryopump. The total clearance of the pump is determined by tests without adsorbant at different temperatures and the volume initially occupied by the zeolite is deducted from it. This gives the effective clearance volume and hence the quantity of gas adsorbed at equilibrium pressure.

We plotted the helium-3 adsorption curves for zeolite 5A at different temperatures as a function of cell pressure (Figure 3). The adsorbed volumes are given in cubic centimetres (s.t.p.) per gram degassed adsorbent. At 20 and 15° K we used 4·7 g zeolite, with hydrogen as coolant. No saturation was detectable in the pressure range studied. Henry's law

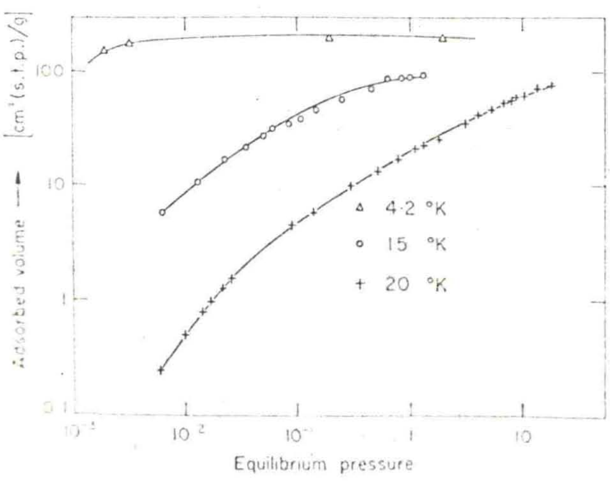

Figure 3. Adsorption of helium-3 at 20, 15, and 4·2° K

$$V = K'P$$

holds good only at low pressures, as was observed by Stern et al. who studied helium-4 adsorption with a different zeolite, 13X. On the other hand, at 4·2° K we obtain a saturation level at 190 cm³(s.t.p.)/g.

We measured the cryopump pumping rate by simply introducing in the preceding circuit a calibrated leak between the tanks and the pump (Figure 2). Only the 'constant flow' method at steady state proved applicable for adsorbents, because a method of decreasing pressure cannot be used in the case of zeolite which does not attain thermal equilibrium quickly enough. The calibrated leak was a very fine orifice in a thin wall. Flow Q is thus proportional to the upstream pressure if this is definitely higher than the pump pressure

$$Q = PC$$

where Q is the flow in torr-litre/second, P = the pressure upstream of the leak in torrs, and C = the leak rate in litres/second. The downstream pump pressure, as read off the Médiovac gauge is p and for the same flow, the pumping rate of the cryopump is

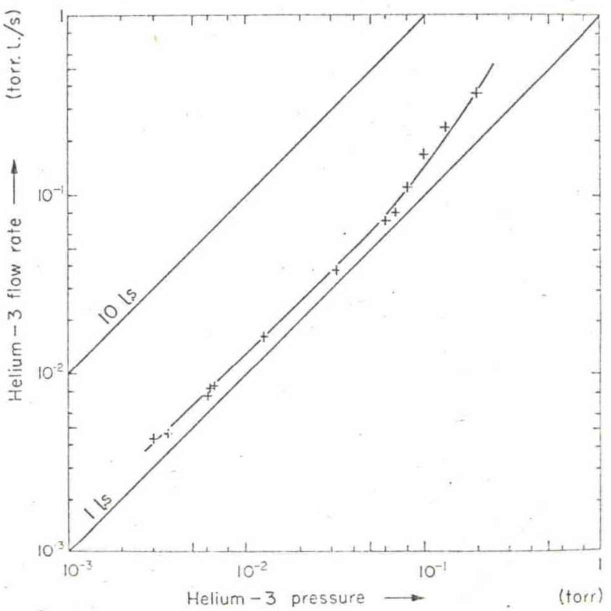

Figure 4. Cryosorption pump at 4·2° K

$$S = \frac{Q}{p}$$

At hydrogen temperatures the pumping rate proved negligible. At 4·2° K we plotted (Figure 4) the pump flow curve as a function of pressure, with 4·7 g of zeolite in the arrangement. In the linear section the pumping rate is 1·3 l./s The effect of saturation becomes evident by a slight decrease of the pumping rate when the adsorbed quantity reaches 100 cm³/g.

On the basis of the above results we designed a cryopump for our cryostat, with greater capacity (adsorption

3 l. of helium-3) and a sufficient pumping rate for [illegible] conditions. For meeting the first requirement we increased the quantity of zeolite. For the second requirement, we tried to provide better thermal contact between [illegible] and zeolite grains, and easier access of the gas to the zeolite by spreading it thinly over a large surface.

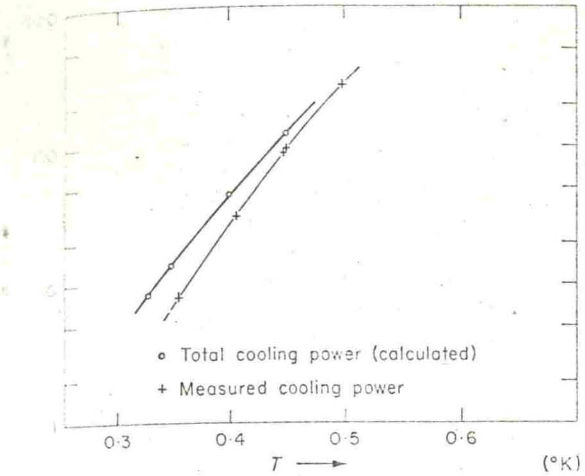

Figure 5. Performance of helium-3 cryostat

The cryopump used is a stainless steel tube of 16 mm diameter with a copper tube extension of the same diameter and 30 cm long. A central channel of copper wire grating, 11 mm across, forms an annular space containing 15 g zeolite. In order to reduce the heat intake of the adsorbant by radiation from outside and facilitate cooling of the pumped gas, a radiation trap was mounted on the tube.

The cryopump is submerged in a helium-4 storage tank which is raised and lowered by means of a hoisting carriage. At the start of a test the helium-3 tanks (0·5 to 2·5 l. s.t.p.) are emptied by means of the cryopump. The storage tank is then lowered, the helium-3 desorbed and it condenses on contact with the helium-4 bath at 1·2° K at its vapour pressure of 20 mm for this temperature.

After closing the pump valve the storage tank is raised, the cryopump quickly reaches thermal equilibrium and the liquefied helium-3 can then be pumped, using a valve for regulating the bath pressure.

The measured pumping rate of the new pump is of the order of 1·7 l./s for a capacity of over 3 l. of helium-3. We reached 0·33° K, at which temperature helium-3 has a vapour pressure of 5 μ. With 2·5 l. of helium-3 the test will last just as long as the helium-4 bath. For measuring the cooling power of the arrangement (Figure 5) we passed into a heating resistor connected to the block a known amount of energy sufficient, at equilibrium, to offset the heat dissipated by vaporization of the helium-3.

During these tests the temperature of the block was measured by means of a carbon resistor calibrated at the laboratory. We also plotted the total theoretical power curve on the basis of the pumping rates of the cryopump (1·7 l./s) and circuits (4 l./s), disregarding the thermal losses.

Conclusion

The preliminary tests have shown the effectiveness of cryosorption pumping of helium-3 at 4·2° K and resulted in a cryostat of simple design and operation. By means of thermal contact and rapid recondensation of the adsorbed gas (the desorption–condensation–cooling process takes less than 10 min) successive cycles for cooling samples of high thermal capacity can be produced with small quantities of helium-3.

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