

## Purification of $^3\text{He}$ from a $^4\text{He}$ Impurity Using Adsorption

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**Abstract**—A method for purification  $^3\text{He}$  gas from a  $^4\text{He}$  impurity using adsorption is described. In terms of productivity and efficiency, this method is on a par with the conventionally used rectification of a liquid  $^3\text{He}$ – $^4\text{He}$  mixture, but is considerably simpler.

High-purity  $^3\text{He}$  is used in many physical experiments. A standard trap cooled with liquid  $^4\text{He}$  easily cleanses  $^3\text{He}$  from all impurities except for the  $^4\text{He}$ . The  $^4\text{He}$  impurity is conventionally eliminated by rectifying a mixture of liquid  $^3\text{He}$  and  $^4\text{He}$  [1–3]. However, this method implies high consumption of the liquid  $^4\text{He}$  and needs equipment that is quite difficult to produce and operate. In this paper, we suggest a new method for  $^3\text{He}$  purification. It is based on the difference in energy of  $^3\text{He}$  and  $^4\text{He}$  adsorption by activated carbon [4, 5]. This method is virtually identical to frontal gas-adsorption chromatography at a liquid-helium temperature (4.2 K).

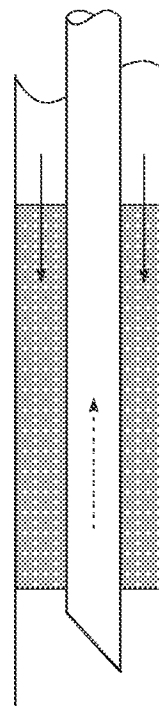
The purification device (Fig. 1) is a chromatographic column that may be inserted in an CTF-10 transport helium Dewar vessel. The column consists of two thin-walled stainless-steel tubes (11 and 3 mm in diameter). The outer tube is blanked-off from the bottom. At the top, both of the tubes are connected to flexible bellow hoses. As a result, the column can easily be removed from the vessel for heating. At the bottom, the space between the tubes is filled with granules of CKT-type activated carbon to a level flush with or above the maximum level of the helium in the transport Dewar vessel. The granules are fixed from top and bottom with brass meshes that are soldered to the inner tube. The total amount of activated carbon is  $12\text{ cm}^3$  (~4 g).

The apparatus for  $^3\text{He}$  purification (Fig. 2) is composed of a needle valve for fine control of the gas stream, a ПТИ-10 mass spectrometer-type leak detector, and a glandless rotary pump or a cryogenic pump (inserted into another transport helium vessel). The pumps were used for pumping of helium gas. The leak detector has been modified for the detection of both  $^4\text{He}$  and  $^3\text{He}$  and is used to measure the composition of a mixture [6].

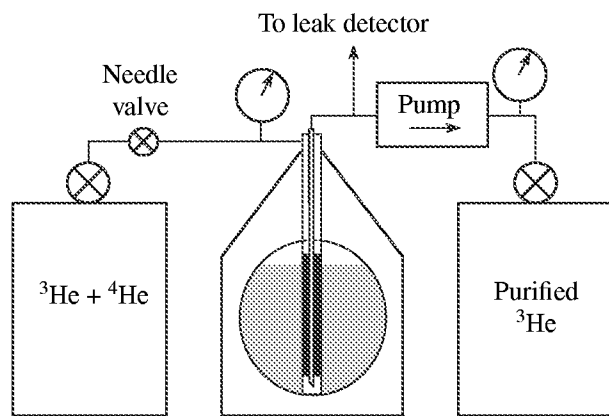
Prior to purification, a certain amount of a  $^3\text{He}$ – $^4\text{He}$  gas mixture is allowed to bleed into the device. The gas-flow rate is regulated with the needle valve. The device is subsequently inserted into a transport Dewar vessel with the liquid helium, and the gas continues bleeding into the device, being adsorbed by the carbon. The valve located at the pump inlet opens upon a significant

increase in the pressure at the device's outlet. The amount of the incoming gas and the flow rate are determined from the pressure in the known volume at the pump outlet. The volume of the adsorbed gas depends only slightly on the composition of the mixture and the pressure (in a range of 5–50 torr); under standard conditions, it is ~1 l.

Preliminary experiments showed that, at pressures in the column of <10 torr, the purification efficiency was higher than at a pressure of ~100 torr. Therefore, the gas purification was carried out at a reduced pressure (it was for this reason that the needle valve was placed at the device's inlet). It was also found that, for a mixture with 18%  $^4\text{He}$ , the initial degree of purifica-



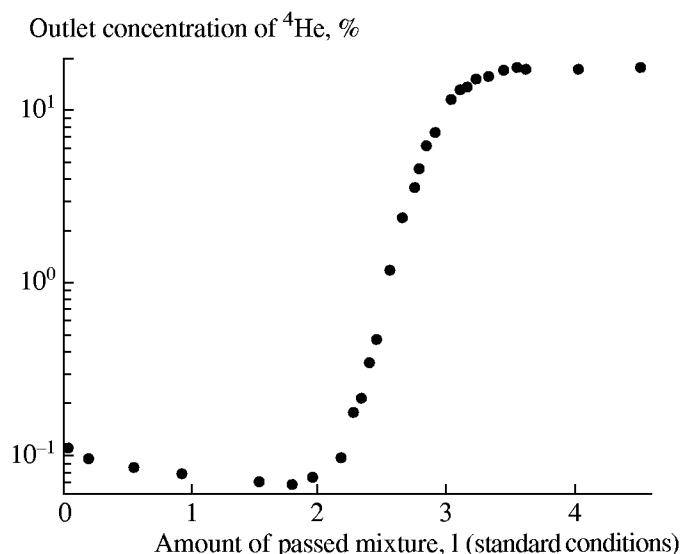
**Fig. 1.** Diagram of the foot of the chromatographic column. The direction of the gas flow is shown with arrows.



**Fig. 2.** Diagram of the apparatus for the separation of  ${}^3\text{He}$  and  ${}^4\text{He}$ .

tion depended on the flow rate, and rates of 0.03 to 0.2 l/min appeared to be optimal. In practice, the flow rate was usually selected from just this range, since the results obtained with the other initial concentrations were good in this case as well. More exact selection of the optimum conditions for gas purification was not done due to its complexity. The equilibrium concentration of the gas over the adsorbate must depend on the pressure and the concentration. Moreover, in a real experiment, the pressure above the carbon not only depends on the flow rate, but also varies throughout the column (e.g., at a flow rate of 0.09 l/min, the pressure drop across the column is  $\sim 10$  torr).

Figure 3 illustrates the dependence of the  ${}^4\text{He}$  concentration in the effluent gas on the volume of the gas passed through the column. The  ${}^4\text{He}$  concentration in the base mixture was 18%. The gas flow rate varied from 0.07 to 0.04 l/min, because the pressure in the vessel with the base mixture decreased. After the passage of a 4.5-l volume of the mixture, the  ${}^4\text{He}$  concentration in the gas adsorbed on the carbon was  $\sim 60\%$ . It is evident that the column is saturated upon passing  $\sim 2$  l of a mixture with this composition. Thereafter, it may be heated, and the adsorbed mixture, enriched in  ${}^4\text{He}$ , may be discharged to another vessel. (This is not shown in



**Fig. 3.** The dependence of the  ${}^4\text{He}$  concentration at the column inlet on the amount of the mixture passed through the column. The base mixture contained 18%  ${}^4\text{He}$ .

Fig. 2.) At smaller  ${}^4\text{He}$  concentrations in the base mixture, more of the mixture may be run through the column before its saturation. In practice, it is more convenient to make the whole of the mixture flow through the column and only then to heat it. The purified mixture may circulate through the column several times until the  ${}^3\text{He}$  purity reaches the required level. The mixture enriched in  ${}^4\text{He}$  may be subsequently cleansed in a separate experiment.

The results from purification a mixture (25 l under standard conditions) with an initial  ${}^4\text{He}$  concentration of 0.1% are presented in the table. The mixture was passed through the column twice; after the first passage, the column was heated, and the adsorbed gas was discharged into an isolated vessel. After purification, it was impossible to measure the concentration of the  ${}^4\text{He}$  admixture in the purified gas, since it was far below the response limit of the leak detector ( $\sim 0.01\%$   ${}^4\text{He}$ ). The overall process of purification took a few hours (in view of the activation of the leak detector, evacuation of the service lines, and leak check of the system). In this case,  $<1$  l of the liquid  ${}^4\text{He}$  evaporated from the Dewar vessel.

Thus, the efficient separation of  ${}^3\text{He}$  and  ${}^4\text{He}$  using adsorption has demonstrated the following advantages of this method over the rectification technique: (1) there is no need for an isolated cryostat with the exhausting of  ${}^4\text{He}$  vapor; (2) significantly lower consumption of the cooling  ${}^4\text{He}$ ; and (3) a simpler design of the apparatus. In addition, this method is comparable to rectification in productivity and efficiency.

**Table**

| Concentration of ${}^4\text{He}$ in the base mixture, % | Amount of mixture passed through the column, l (standard conditions) | Concentration of ${}^4\text{He}$ in the purified mixture, % | Concentration of ${}^4\text{He}$ in the adsorbed gas, % |
|---|--|---|---|
| 0.1   | 24   | $\sim 0.02$   | 2.2   |
| $\sim 0.02$   | 23   | $< 0.01$  | 0.5   |

## ACKNOWLEDGMENTS

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