

Separation of ^3He from ^3He - ^4He Mixture by means of Adsorption

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A simple technique for purifying ^3He from a ^4He admixture is presented. The technique is based on different adsorption energies of ^3He and ^4He .

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1. INTRODUCTION

The purity of commercially available ^3He is often not high enough for physical experiments. Also after certain experiments ^3He becomes impure. All impurities but ^4He can be readily disposed of by using a trap cooled with liquid ^4He . The ^4He admixture is commonly eliminated using rectification method ¹⁻³. This method requires condensation of the ^3He - ^4He mixture and consequently a ^4He evaporation cryostat utilization. We propose a simpler method based on the difference in adsorption energies of the helium isotopes on activated charcoal. This method is in fact a form of frontal chromatography carried out at a temperature of liquid ^4He . Part of the results presented below were published in Ref. 4.

2. APPARATUS

The sketch of the apparatus is shown in Fig. 1. The main parts of it are the chromatographic column (1), a needle valve (2), a hermetic rotary pump with the valve (3) at the inlet and tanks for gas. A mass-spectrometer type leak detector was used to measure the composition of the ^3He - ^4He mixture.

The chromatographic column is designed as follows. A 3 mm outer diameter stainless tube is inserted into an 11 mm outer diameter stainless tube. The latter is blanked off at the lower end and at the top two flexible bellow hoses are welded to the tubes, so the column can be easily put into

and out of the liquid helium storage dewar (4 in Fig. 1): the diameter of the outer tube is chosen to fit the standard 10 litres storage dewar STG-10. The space between the tubes is packed with activated charcoal up to the highest possible level of liquid ^4He in the dewar. The total amount of the charcoal is 12 cm^3 (about 4 g).

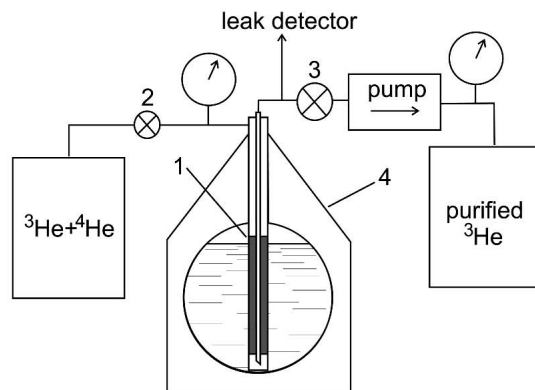


Fig. 1. The sketch of the apparatus. 1 – the chromatographic column, 2 – the needle valve which is used for fine adjustment of the flow rate, 3 – the valve at the inlet of the rotary pump, 4 – liquid helium storage dewar.

3. OPERATION AND RESULTS

The needle valve was placed at the inlet of the column because it was found that the purification efficiency is higher at pressures in the column of <10 Torr than at pressure of ~ 100 Torr. Optimal flow rates proved to be in range of 0.03-0.2 l/min, so, before the purification starts, a required gas flow rate is set using this valve. Then the column is inserted into the liquid ^4He storage dewar, the valve (3) is opened and the purification starts. The concentration of ^4He at the outlet of the column can be then continuously controlled by the leak detector. A certain amount of gas is adsorbed on the charcoal surface before the pressure at the inlet of the pump starts rising. This amount only slightly depends on the mixture composition and on the pressure and for our column is about 1 litre of gas at standard conditions.

In Fig. 2 are shown the measured dependencies of ^4He concentration at the outlet of the column on the volume (at STP) of the gas passed for two different initial ^3He - ^4He mixtures. It is seen that at the beginning the outflowing gas contains much less ^4He than in the initial mixture. But, after

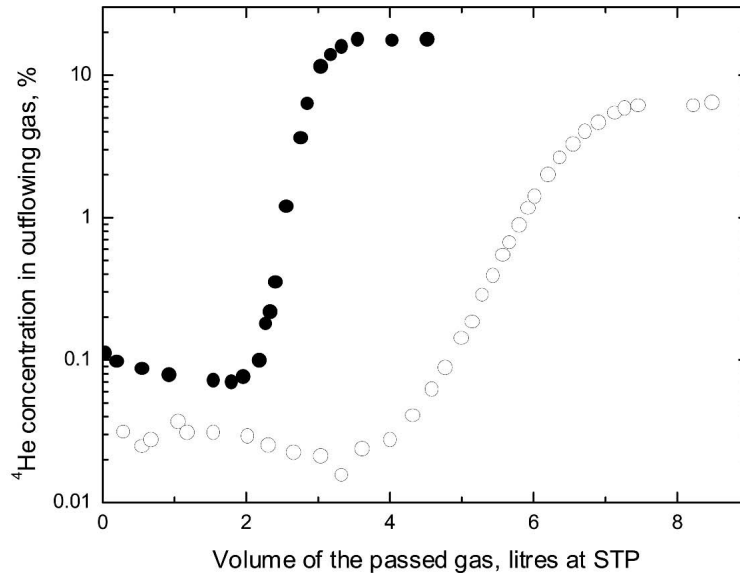


Fig. 2. ^4He concentration at the column outlet as a function of the gas volume passed through the column. The initial concentration of ^4He in the mixture was: ● - 18%; ○ - 6.5%. The flow rate was in range 0.04 – 0.1 l/min.

the passage of some amount of the mixture, the column is saturated and the concentration of ^4He reaches the concentration at the inlet of the column. The peculiarity of these dependencies is that before the saturation occurs the ^4He concentration at the output slightly decreases as the gas passes. One possible explanation for this effect is that there is some optimal pressure at which the difference between the compositions of the adsorbed gas and the passing gas is maximal. As the gas flows from the " $^3\text{He}+^4\text{He}$ " tank, the pressure in the tank drops as well as the pressure gradient through the column. Correspondingly, the concentration at the column outlet can change with time. We did not study this ^4He concentration decrease effect in detail, however it was found that the effect is more prominent at low flow rates.

In principle, when the saturation starts, the purification process may be stopped. Then the column can be taken out of the storage dewar and heated up in order to pump out the adsorbed mixture (enriched with ^4He) into another tank for "dirty" helium (not shown in Fig. 1). However, in practice, it is more convenient to allow the whole available amount of the

mixture to pass through the column and only then to heat up the column. The purified mixture can be then passed through the column one more time and so on, until the purity of ^3He reaches the required value. In the table below we present the results of such a procedure. 25 litres of the mixture with the initial ^4He concentration of 0.1% were passed through the column twice and finally we have got about 23 litres of purified gas and about 2 litres of gas enriched with ^4He . After the second passage we were not able to measure the concentration of ^4He admixture, since it was well below the sensitivity limit of our leak detector ($\sim 0.01\%$ of ^4He). Also, the precision of our measurements was not high enough to deduce the final ^4He concentration value from the known values of ^4He concentration in the adsorbed gas and in the initial mixture. The overall process of purification took a few hours and cost less than 1 litre of liquid ^4He evaporated from the storage dewar.

Initial concentration of ^4He , %	Volume of the passed gas, litres at STP	Final concentration of ^4He , %	Concentration of ^4He in the adsorbed gas, %
0.1	~ 24	~ 0.02	2.2
~ 0.02	~ 23	< 0.01	0.5

We can conclude that (at least down to 0.01% ^4He concentration) the purification of ^3He can be effectively done using the simple technique described above. We believe that this method also works well for lower concentrations.

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