Investigations regarding helium isotopes separation by gas chromatography

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Introduction

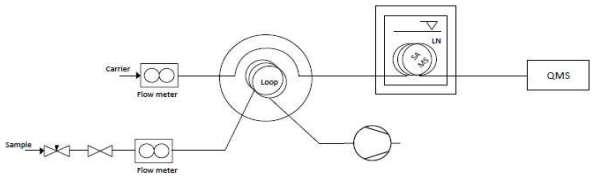
Helium-3 is a rare isotope of helium. It is used in neutron detectors because it has a high cross-section for absorption of thermal neutrons, in achieving very low temperature of few mK. It can also be used in fusion reactions by either deuterium or tritium, as it produces a much lower neutron flux than deuterium-tritium fusion reaction.

The multiple economic domains that involve the use of He-3 have made it a gas of great importance and its separation from different gas mixtures has become a priority in the research world. The main source of He-3 is the decay of tritium, the radioactive isotope of hydrogen that undergoes beta decay with a half-life of 12,3years.

The separation of helium 3 from helium 4 was obtained by various methods: heat flush method followed by distillation, super-leak, and distillation, or super-leak followed by thermal diffusion, all involving liquid phase of helium. Our proposed method is based on the separation of atoms from mixtures using microporous materials, in gaseous phase. We use the chromatographic method for the investigation and evaluation of the separation and recovery of helium isotopes. The chromatographic column used for this study is a 5 meters long, packed column filled with 5A molecular sieve, using hydrogen as carrier gas. The column is operated at 77 K and at different carrier gas flow rates in the range of 20 – 90 standard cubic centimeters per minute (sccm).

$$^{3}_{1}H \rightarrow {}^{3}_{2}He + e^{-} + \bar{\vartheta}$$

Experimental setup



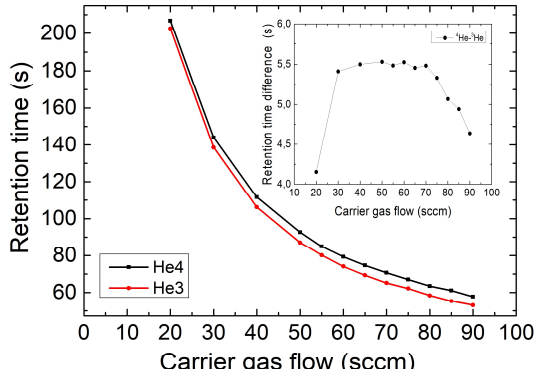
Schematic diagram of the experimental setup

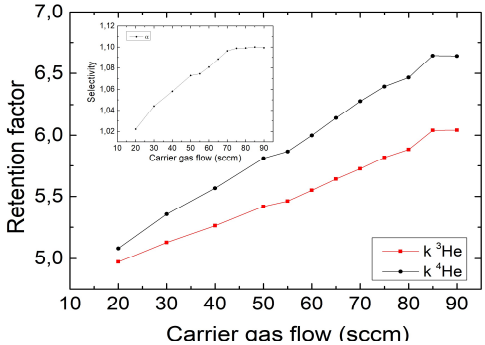
Experimental procedure

Two sets of measurements were performed, one for helium 4 and one for helium 3, using hydrogen as carrier gas, at flow rates in the range of 20 – 90 sccm. For each measurement, retention time, retention factor, full width at half maximum, selectivity, resolution were determined using Gauss fit.

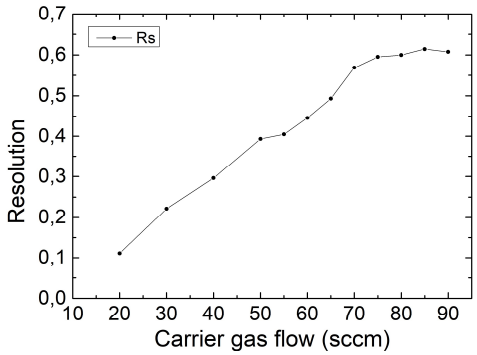
- The chromatographic column is submerged in liquid nitrogen in a Dewar vessel and the carrier gas flow is set to the desired value;
- The sample loop, with the VALCO valve in LOAD position, is cleaned using a vacuum pump to remove the carrier gas from the previous run. We chose this approach instead of purging with sample gas because we didn't want to waste it;
- After this step, the sample is injected in the loop, monitoring the flow in order to obtain the established quantity of gas (1ml at 101kPa);
- The next step is to switch the valve into INJECT position allowing the sample to be transported with the carrier gas stream through the chromatographic column to the QMS;
- At the exact time of the switch, the analysis on the QMS is started and the spectrum for the interest m/z (3 or 4, in function of which helium isotope is fed in the loop) is recorded;
- This procedure is repeated for each of the two gases and for different flow rates of the carrier gas that are studied.

Results and discussion





Retention factor versus carrier gas flow rates



Resolution versus carrier gas flow rates

In our investigation and evaluation of the separation and recovery of helium isotopes by gas chromatographic method, we observed a difference between the retention times measured for the two helium isotopes. By using Gaussian peaks fit we determined the separation parameters: retention time, retention factor, full width at half maximum, selectivity, and resolution.

Using the experimental data we established the optimum operation parameter and proposed a method for improving the resolution.

Isotope	F(sccm)	t_r (s)	FWHM (s)	k	α	R_s
^{3}He	75	61.688	5.148	5.818	1.098	0.595
^{4}He		67.020	5.396	6.393		

Acknowledgements

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