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The separation of nitrogen isotopes by gas chromatography

Gas–solid chromatography has been successfully used to separate isotopic mixtures of permanent gases, such as oxygen and methane isotopes[1,2]. A calculation of the separation factor for nitrogen isotopes from unresolved peaks has been reported by SIDEMAN AND GILADI[3]. In this note we report the results obtained in an attempt to separate nitrogen isotopes.

A mixture of $^{14}N_2$ and $^{15}N_2$ was prepared by alkaline fusion of $^{15}NH_4NO_3$ and successive oxidation of the resulting ammonia. The gas chromatograph consisted of an etched glass capillary column (175 m, 0.28 mm I.D.), cooled in a Dewar with liquid nitrogen and a thermal conductivity microcell (Gow Mac JDC 301). The preparation of the column has been previously described[1]. The carrier gas was a mixture of 55% He and 45% CO. This was necessary for deactivating the column surface with a suitable layer of adsorbed molecules.

Previous experiments have shown that carbon monoxide gives the best results here.

Attempts were made to use oxygen and methane as deactivants, but tailed peaks were always obtained with these two gases. Carbon monoxide is a more polar molecule and its deactivating power is stronger.

When the temperature of the above mixture is 77°K, the value of P/P_0 for CO (P = partial pressure, P_0 = vapour pressure at liquid nitrogen temperature) is about 1; therefore, we can assume that all the active centers in the silica layer are covered with a multi-layer of CO molecules. Under these conditions the chromatographic peaks of nitrogen are fully symmetrical.

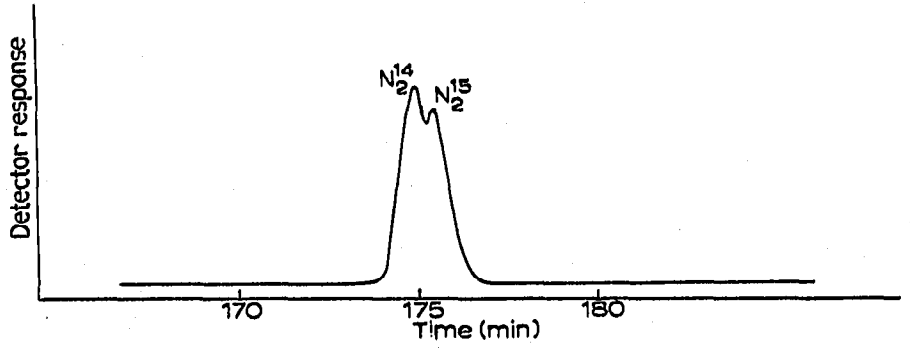

Fig. 1. Separation of a mixture of $^{14}N_2$ and $^{15}N_2$ in an etched glass capillary column (175 m, 0.28 mm I.D.). Carrier gas: He–CO (45%); pressure, 13 cm Hg; temperature, 77 °K; flow rate, 0.6 ml/min.

The separation of $^{14}N_2$–$^{15}N_2$ is shown in Fig. 1. Unfortunately, under such conditions the capacity ratio is too small (175 min is the retention time including the dead time of the column of 77 min, then the capacity ratio is 1.25) and consequently, the maximum resolving power of the column cannot be completely realised.

The separation factor is 1.006, while the ratio of the vapour pressures taken from the literature[4] is 1.008.

At a lower temperature, where higher capacity ratios can be obtained, a better separation is expected, because the column operates at the maximum efficiency.

These results do show that gas chromatography can be employed to separate nitrogen isotopes.

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