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Separation of Hydrogen Isotopes Mixtures Using Different Concentrations of Gas Mixtures by Gas Chromatography

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In this paper, method gas chromatography for the analysis of the different concentrations of gas mixtures containing hydrogen isotopes. The method described in this paper was based on using a capillary molecular sieve 5A column which has been operated at 174 K. The isotopic species of hydrogen are: H_2, D_2, T_2, HD, HT, DT, *ortho*-H2, *para*-H2, *ortho*-D2 or *para*-D2, where D stands for ^2H and T for ^3H. In all cases, the *p*-H2 (*para*-H2) form appears before the *ortho*- form. These forms are presented in our chromatograms. We used a gas mixture with the next isotopic species of hydrogen H_2, D_2, HD, *ortho*-H2, *para*-H2. *Ortho*- and *para*-deuterium are not so easily separated. In this way *ortho*-deuterium appearing first. This paper discusses about the specific design of the gas chromatograph and presents the chromatograms measured for different concentrations of isotopic species of hydrogen gas mixture.

Key Words: Gas chromatography, Isotopic species of hydrogen, Pulsed discharge helium ionization detector, 5 Å Molecular sieve.

INTRODUCTION

Gas chromatography (GC) is a well-established analytical tool for the determine of a large variety of gas compositions. Commercial separation columns and special detectors are available for almost any gas analytical application. Gas chromatographic separation of hydrogen isotopes have been reported in the literature dating from the late 1950's. Basically, three approaches have been employed to effect separations on an analytical scale and these approaches may be distinguished on the basis of the column packing material used.

For the separation of hydrogen isotope mixture special gas chromatograph have been developed capable of separating and analyzing quantitatively the isotopic species of hydrogen[1]. The quantitative analysis of hydrogen isotopes is of great importance of separating processes of hydrogen isotopes[2].

In this paper, the specific design of the gas chromatography and the chromatograms measured for different concentrations of isotopic species of hydrogen gas mixture are discussed.

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EXPERIMENTAL

The gas chromatograph employed in this work was a type 3800 from Varian Analytical Instrument. The Varian 3800 gas-chromatograph is equipped with a capillary molecular sieve 5A column with following characteristics: (i) the length of the gas chromatography column is: 50 m; (ii) the inside diameter of the GC column is: 0.32 mm; (iii) the film thickness of the GC column is: 30 µm.

The operating temperature of the GC column was -99 ºC. The temperature of the oven of the GC column was maintained in the range of 0 to -99 °C, by spraying liquid nitrogen into the oven. A temperature controller to control the liquid nitrogen flow and the heater was used.

As detector, a Pulsed Discharge Helium Ionization Detector (PDHID) was used. The carrier gas used was helium (99.999 % purity). The sample loops: 5 µL. The GC column was conditioned before to use this for the separate of the hydrogen isotopes mixtures.

RESULTS AND DISCUSSION

The method described in this paper, was based on using a capillary molecular sieve 5A column which has been operated at only 173 K. The method used was calibrated with standard gas of protium and deuterium by external standard calibration type. The retention times were relatively short, about 10-12 min and the result is a good separation of *para*-H_2, *ortho*-H_2, HD, D_2.

The sample in the 5 mL sampling volume is injected into system which uses helium as carrier gas. The carrier flow rate was 3.7 mL/min at a pressure of 10 psi.

The GC column was conditioned before use for the separation of the hydrogen isotopes mixtures. A small flow of helium (1.2 mL/min) was maintained through the column during conditioning.

For the analysis, 5 gas mixtures of the hydrogen isotopes mixtures were used. The gas mixtures had different concentrations. The concentrations of the hydrogen isotopes species can be observed in the next chromatograms.

In Fig. 1, the chromatogram showed the concentrations of hydrogen isotopes species at 11.371 % *p*-H2; 87.326 % *o*-H2; 0.182 % D2.

Fig. 2 showed the chromatograms obtained after analysis of gas mixtures: (i) in the '1' chromatogram are showed that the concentrations of hydrogen isotopes species are 15.223 % *p*-H2; 73.267 % *o*-H2; 0.682 % HD; 8.706 % D2, (ii) in the '2' chromatogram are showed that the concentrations of hydrogen isotopes species are 12.248 % *p*-H2; 60.637 % *o*-H2; 0.716 % HD; 26.378 % D2. (iii) in the '3' chromatogram are showed that the concentrations of hydrogen isotopes species are 9.326 % *p*-H2; 40.543 % *o*-H2; 2.598 % HD; 47.533 % D2, (iv) in the '4' chromatogram are showed that the concentrations of hydrogen isotopes species are 0.411% *p*-H2; 1.844 % *o*-H2; 0.571 % HD; 97.174 % D2. All these results are presented in the Table-1.

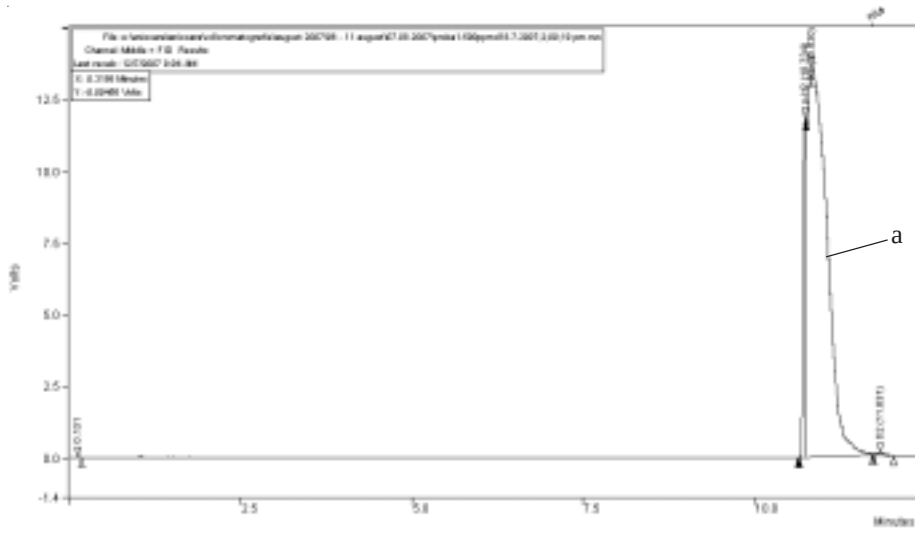

Fig. 1. Sample - 11.371 % *p*-H2; 87.326 % *o*-H2; 0.182 % D2

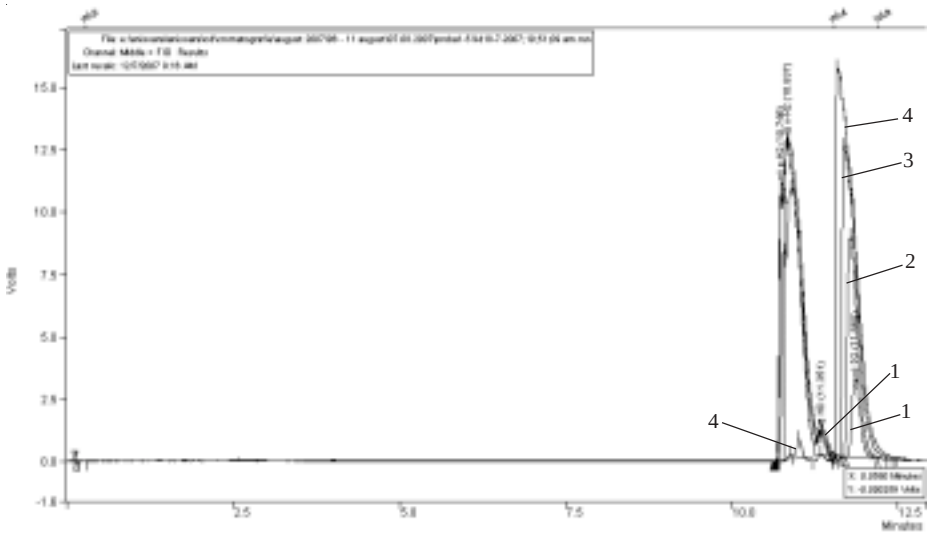

Fig. 2. Diagram of all chromatograms obtained after analysis of gas mixtures

TABLE-1
RESULTS OF ANALYSIS

Chromatograms	% *p*-H2	% *o*-H2	HD	D2
'a'	11.371	87.326	-	0.182
'1'	15.223	73.267	0.682	8.706
'2'	12.248	60.637	0.716	26.378
'3'	9.326	40.543	2.598	47.533
'4'	0.411	1.844	0.571	97.174

In all these chromatograms, a separation between *para*- and *ortho*-hydrogen forms has been observed.

This analysis was also performed using different concentrations. Conclusively, the chromatograms presented in this paper demonstrate a good separation performance for capillary molecular sieve 5A column at only 174 K, in case of hydrogen species and particularly for the *para*-H2 and *ortho*-H2 forms.

In the chromatograms (Figs. 1 and 2), it is observed that the retention times were relatively short and same for all hydrogen isotopes species.

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