Journal of Chromatography, 128 (1976) 163–165

CHROM. 9493

Note

Use of a nematic liquid crystal for the gas chromatographic separation of aza-heterocyclic compounds

M. PAILER and V. HLOŽEK*

Institute of Pharmaceutical Chemistry, University of Vienna, Vienna (Austria)

(First received May 10th, 1976; revised manuscript received July 2nd, 1976)

Many publications have been concerned with detection and separation of aza-heterocyclic compounds. These compounds, several of which are known to be carcinogenic, have been found in cigarette smoke[1,2], air pollutants[3] and car exhaust fumes[4].

The separation of these aza compounds has been attempted by thin-layer chromatography[5,6], gas chromatography[7], paper chromatography[8] and high-performance liquid chromatography[9,10]. It is well known that excellent separations of polyaromatic hydrocarbons (up to five fused rings) (PAHs) are possible by using nematic liquid crystals as stationary phases for gas–liquid chromatography. Baseline separations for a wide range of PAH compounds have been reported by Janini *et al.*[11].

EXPERIMENTAL

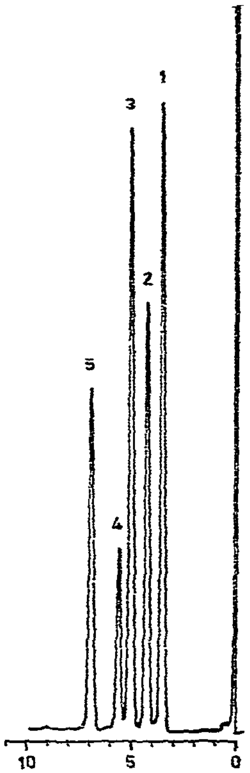
N,N'-Bis(*p*-methoxybenzylidene)-α,α'-bis-*p*-toluidine (BMBT) (Eastman-Kodak, Rochester, N.Y., U.S.A.) was employed as the stationary phase. The transition points were 181° (solid–nematic) and 320° (nematic–isotropic). The packing material, 2.5 and 10% (w/w) on Chromosorb W AW DMCS, 100–120 mesh, was prepared by using a solution of a weighed amount of BMBT in freshly distilled chloroform. The length of the Pyrex glass columns used was 1.4 m with an I.D. of 0.2 cm. The columns were conditioned for 24 h at 265° and a low carrier gas flow-rate (10 ml/min) before use.

The chromatograms (Figs. 1–3) were obtained by means of a Varian Aerograph Series 2100 instrument equipped for dual column analysis, utilizing flame-ionization detectors connected with a Goerz Servogor S strip-chart recorder (paper speed for all chromatograms 0.5 cm/min).

The following conditions were employed: injector temperature, 250°; detector temperature, 260°; temperature programme for column oven: (a) 200–265° at 8°/min; (b) 190–265° at 6°/min; and (c) 190–265° at 8°/min. The nitrogen carrier gas flow-rate was measured with a soap-bubble flow meter (40 ml/min).

---

[*] To whom correspondence should be addressed.



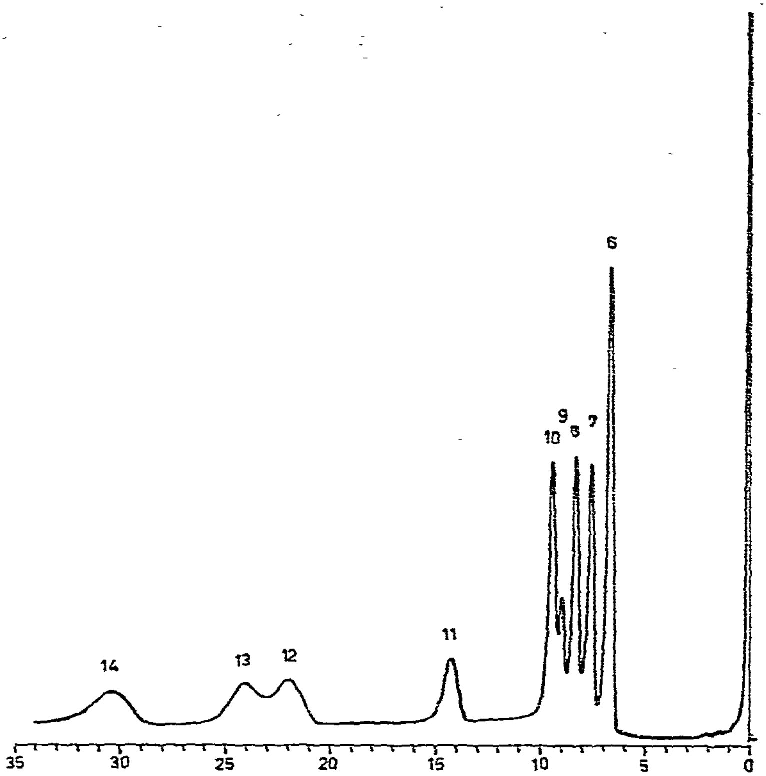


Fig. 1. Separation of polynuclear aza-heterocyclic compounds using 10% BMBT on Chromosorb W AW DMCS, 100–120 mesh as stationary phase. 1 = Phenazine (0.3 μg); 2 = benzo[*h*]quinoline (0.2 μg); 3 = acridine (0.3 μg); 4 = benzo[*f*]quinoline (0.1 μg); 5 = carbazole (0.2 μg). Temperature programme: (b).

Fig. 2. Separation of polynuclear aza-heterocyclic compounds using BMBT (2.5% BMBT on Chromosorb W AW DMCS, 100–120 mesh) as stationary phase. 6 = Benzo[*c*]acridine (0.2 μg); 7 = benzo[*a*]acridine (0.1 μg); 8 = 7-methylbenzo[*c*]acridine (0.1 μg); 9 = isomer of 8 (unknown amount); 10 = 5,7-dimethylbenzo[*a*]acridine (0.1 μg); 11 = dibenzo[*a,c*]acridine (0.1 μg); 12 = dibenzo[*a,j*]-acridine (0.2 μg); 13 = dibenzo[*a,h*]acridine (0.2 μg); 14 = dibenzo[*a,i*]carbazole (0.2 μg). Temperature programme: (a).

RESULTS

The aza-heterocyclic compounds separated are listed in the legends of the figures according to their retention times. They were dissolved in acetone and 1 μl of the solutions were injected.

Good separations and high symmetry of the peaks as well as the relative independence of the amount of stationary phase used are the advantages of this method. The high thermal stability and the unusually wide range of the nematic state (181–320°) of the stationary phase permits the complete separation of tri-, tetra- and pentacyclic compounds. The aza-heterocyclic compounds used differ not only in their dimensions (length-to-breadth ratio) but also in their polarity and basicity. Each of these properties has a significant influence on the retention time.

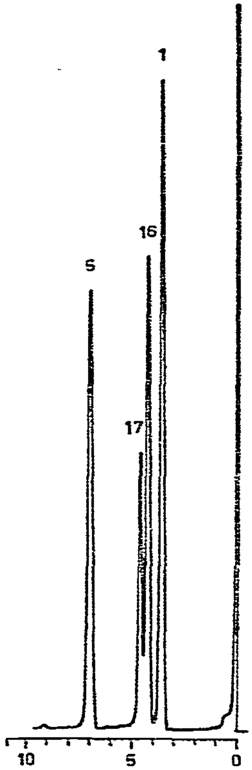


Fig. 3. Separation of polynuclear aza-heterocyclic compounds using BMBT (10% BMBT on Chromosorb W AW DMCS, 100–120 mesh) as stationary phase. 1 = Phenazine (0.3 μg); 16 = N-ethylcarbazole (0.2 μg); 17 = N-vinylcarbazole (0.1 μg); 5 = carbazole (0.2 μg). Temperature programme: (c).

ACKNOWLEDGEMENTS

The authors thank Dr. H. Klus (Austria Tabak Werke, Vienna) for the samples of the compounds studied and Mr. W. Deimbacher for preparing the columns.

REFERENCES

1 B. L. van Duuren, J. A. Bilbao and C. A. Joseph, *J. Nat. Cancer Inst.*, 25 (1960) 53.
2 R. L. Stedman, *Chem. Rev.*, 68 (1968) 153.
3 E. Sawicki, J. E. Meeker and M. J. Morgan, *Int. J. Air Water Pollut.*, 9 (1965) 291.
4 E. Sawicki, J. E. Meeker and M. Morgan, *Arch. Environ. Health*, 11 (1965) 773.
5 E. Sawicki, T. W. Stanley, J. D. Pfaff and E. C. Elbert, *Anal. Chim. Acta*, 31 (1964) 359.
6 E. Sawicki, T. W. Stanley and W. C. Elbert, *J. Chromatogr.*, 18 (1965) 512.
7 G. Alberini, V. Cantuti and G. P. Cartoni, in A. B. Littlewood (Editor), *Gas Chromatography*, Institute of Petroleum, London, 1967, p. 328.
8 M. Lederer and G. Roch, *J. Chromatogr.*, 31 (1967) 618.
9 S. Ray and R. W. Frei, *J. Chromatogr.*, 71 (1972) 451.
10 R. Vivilecchia, M. Thiebaud and R. W. Frei, *J. Chromatogr. Sci.*, 10 (1972) 411.
11 G. M. Janini, K. Johnston and W. L. Zielinski, *Anal. Chem.*, 47 (1975) 670.