Notes

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Separation of polynuclear aromatic hydrocarbons by gas–solid chromatography on graphitized carbon black deposited on Chromosorb W

Much attention has been given to the analysis of polynuclear aromatic hydrocarbons (PAH) and especially to analysis by chromatographic methods[1]. In procedures for the separation of PAH by gas–solid chromatography (GSC) on alkali and alkaline earth chlorides[2–10], on other inorganic salts[3,7,11–13], on graphitized carbon black[9,14–16], or on silica[17], the much higher thermal stability and different separating properties of these adsorbents in comparison with those of gas–liquid chromatography (GLC) have been emphasized. The difficult elution of PAH on graphitized carbon black in conventionally packed columns[14] has been facilitated by depositing a thin layer of graphitized carbon black on the walls of capillary columns[15] or on the inert porous support[16].

After our recent success[16] in the GSC separation of phenanthrene, anthracene and carbazole, using graphitized carbon black deposited on Chromosorb W we have tried this adsorbent on some PAH with four and five condensed rings.

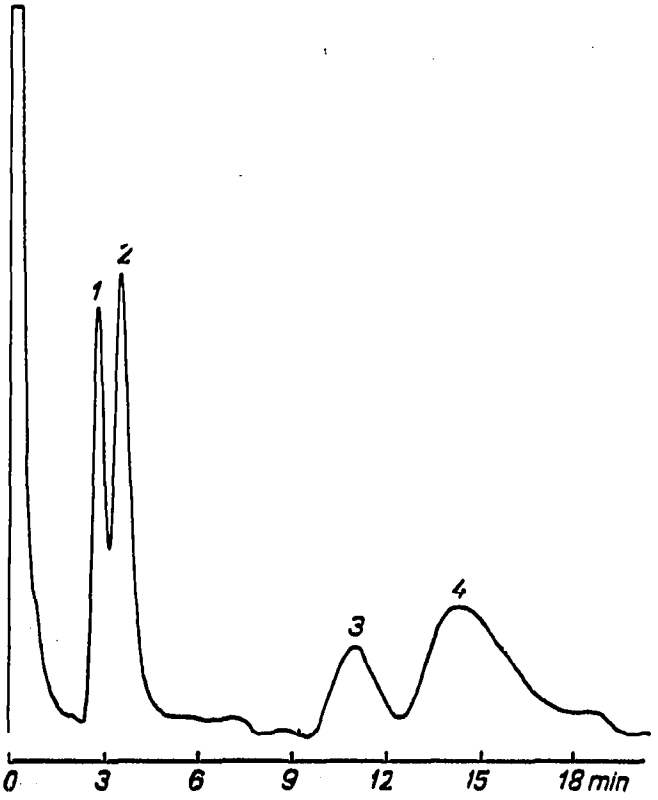

Fig. 1. Chromatogram of pairs of isomeric PAH on the column with graphitized carbon black on Chromosorb W (15:85) at 460°. Other conditions in text. 1 = Benzo[*e*]pyrene, 2 = benzo[*a*]-pyrene, 3 = dibenz[*a*, *c*]anthracene, 4 = dibenz[*a*, *h*]anthracene.

Experimental

Instrument. A gas chromatograph Fractovap GV (Carlo Erba, Milano, Italy) with a flame-ionization detector (FID) connected with an EZ2 recorder (Laboratory Instruments N.E., Prague, Czechoslovakia).

Column. A stainless-steel tubing 95 cm long, 2 mm. I.D., U shaped, coated with 15 % of graphitized carbon black (a thermal carbon black TeG-10 specimen produced in the U.S.S.R. graphitized at 2200° for 8 h by Elektrokarbon N.E., Topol'čany, Czechoslovakia; spec. surface 7.9 m^2/g) deposited on 85 % of Chromosorb W (80–100 mesh, supplied by Carlo Erba) from a suspension in benzene. The amount of the filling in the column was 1.28 g, the content of the graphitized carbon black 192 mg.

Conditions. Column temperature, 460°; injector temperature, 400°; detector temperature, 400°. Carrier gas (argon) flow rate, 20 ml/min; inlet pressure, 2.8 atm. Samples (0.1–0.4 μl) of 0.1–0.5 % benzene solutions of PAH (0.1–2.0 μg of PAH) were injected with a Hamilton 10 μl microsyringe.

The results of the separations are presented in Table I and in Fig. 1.

TABLE I

RELATIVE RETENTION VOLUMES OF POLYNUCLEAR AROMATIC HYDROCARBONS ON THE COLUMN WITH GRAPHITIZED CARBON BLACK ON CHROMOSORB W (15:85) AT 460°

Hydrocarbon	V_R
Fluoranthene	0.07
Pyrene	0.07
Benzo[*b*]fluorene	0.12
Chrysene	0.23
Benz[*a*]anthracene	0.25
Benzo[*e*]pyrene	0.79
Perylene	0.81
Benzo[*b*]fluoranthene	0.92
Benzo[*k*]fluoranthene	0.99
Benzo[*a*]pyrene	1.00[a]
3-methylcholanthrene	1.9
Dibenz[*a*, *c*]anthracene	3.1
Benzo[*g*, *h*, *i*]perylene	3.7
Dibenz[*a*, *h*]anthracene	4.1

[a] 3.4 min.

Discussion

On the graphitized carbon black packed column (70 cm × 2,3 mm) at 470°, benz(*a*)anthracene and chrysene are not eluted after 2 h[14]; whereas on our column they appear within 50 sec (Table I). We use a temperature 125° lower than that used with the capillary column coated with a thin layer of graphitized carbon black[15], and the peaks are still symmetrical (Fig. 1).

The separation factors for benzo[*a*]pyrene/benzo[*e*]pyrene and dibenz[*a*, *h*]anthracene/dibenz[*a*, *c*]anthracene on graphitized carbon black (Table I) are markedly higher than those on organic phases[18–21] or on alkali and alkaline earth chlorides[5,10]. In order to separate these pairs by GLC it usually is necessary to use capillary columns[18,22], but in our case the short packed column is satisfactory (Fig. 1).

Results

Graphitized carbon black on Chromosorb W (15:85) can be used for the fast GSC separation of PAH. This adsorbent makes possible the separation of benzo[*e*]-pyrene from benzo[*a*]pyrene and dibenz[*a*, *c*]anthracene from dibenz[*a*, *h*]anthracene on a column 95 cm long, 2 mm I.D. at a temperature of 460°.

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Research Institute of Coal Tar Chemistry, J. FRYČKA
Urxovy závody N.E., Valašské Meziříčí (Czechoslovakia)

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