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Note

Capillary gas chromatography of the isomeric dimethylnaphthalenes and of some additional aromatic compounds

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Attempts to separate the dimethylnaphthalenes by capillary and packed column gas chromatography have been summarised recently by Tesařík *et al.*[1]. Additional retention data are available[1–4]. An examination of all ten isomers using the silicone stationary phases OV-17 and OV-275 does not, however, appear to have been made. Retention indices of all ten dimethylnaphthalenes and of certain selected aromatic compounds on open tubular glass capillary columns that have been wall-coated with OV-17 and with OV-275 are now reported.

EXPERIMENTAL

Wall-coated open tubular glass columns (25 m) were obtained from Chrompack: 0.27 mm I.D. 0.16 μm OV-17 and 0.28 mm I.D. 0.15 μm OV-275. They were used in a Perkin-Elmer F33 gas chromatograph equipped with a flame ionisation detector. Helium (12 p.s.i., *ca.* 1 ml/min) was used as the carrier gas.

Volumes of 1 μl of solutions containing 0.01 % (w/v) of aromatic compounds were injected into the heated (175°) injection port, which was equipped with a gas-stream splitter (100:1). The columns were temperature programmed from 60–160° at 2°/min. Appropriate alkanes were incorporated into test solutions in order to obtain retention indices, which were calculated using a form[5] of the equation derived by Van den Dool and Kratz[6] for conditions of linear temperature programming:

$$RI_{(x)} = 100 \cdot \left(\frac{T_{r(x)} - T_{r(z)}}{T_{r(z+1)} - T_{r(z)}} + z \right)$$

where $RI_{(x)}$ = retention index of compound x
T_r = retention temperature
z and $z+1$ = normal alkanes with z and $z+1$ carbon atoms respectively.

The separation number, or Trennzahl (TZ), was used as an indicator of column efficiency. The C_{11}–C_{12} fatty acid methyl esters were selected as test compounds for this purpose since the commonly used *n*-alkanes chromatograph inefficiently on OV-275. Peaks were quantified by measurement of their areas (peak height × width at half height).

RESULTS AND DISCUSSION

The retention indices obtained are listed in Table I (in order of boiling point).

Although the OV-17 column used had a satisfactory TZ of 32, the less efficient (TZ = 11–13) OV-275 column had more potential for use in dimethylnaphthalene analysis, especially in the separation of the less votatile isomers.

TABLE I

BOILING POINTS AND RETENTION INDICES OF COMPOUNDS STUDIED

Compound	*Boiling point** (°C; 760 mm)*	*Retention index***	
		OV-17	*OV-275*
Naphthalene	218	1331	1762
Benzo[*b*]thiophene	221	1354	1818
2-Methylnaphthalene	241	1440	1851
1-Methylnaphthalene	245	1463	1881
Biphenyl	255	1545	1973
Diphenyl ether	258	1576	1940
2,6-Dimethylnaphthalene	262	1547	1939
2,7-Dimethylnaphthalene	262	1547	1939
1,7-Dimethylnaphthalene	263	1568	1973
Diphenylmethane	264	1601	2011
1,3-Dimethylnaphthalene	265	1571	1966
1,6-Dimethylnaphthalene	265	1571	1973
1,4-Dimethylnaphthalene	268	1596	1995
2,3-Dimethylnaphthalene	268	1591	2026
1,5-Dimethylnaphthalene	269	1600	2004
1,2-Dimethylnaphthalene	271	1614	2045
3-Methylbiphenyl	273	1652	2063
1,8-Dimethylnaphthalene	277	1646	2097
Acenaphthene	278	1664	2120
Dibenzofuran	287	1695	2237
Fluorene	295	1773	2344

* Average of values given in some or all of ref. 7–10.

** As defined in Experimental. Average of 3–4 values. Subsequently, a reduction of about 10 index units was observed for most compounds on OV-275. This effect is presumably due to column bleed. Baseline separation is obtained between peaks about 4 and 8 index units apart on OV-17 and OV-275 respectively. Little useful separation is obtained between peaks whose retention indices differ by less than half of these values.

Relative response factors obtained from the OV-275 column had an overall relative standard deviation of $\pm 5.4\%$ (21 degrees of freedom).

It is known[7] that Bentone 34 selectively retards certain dimethylnaphthalene isomers, and so a judicious mixture of OV-275 and Bentone 34 might yield a separation comparable with that obtained by Tesařík *et al.*[1], who employed two dual-phase glass capillary columns in series and even so failed to effect a completely satisfactory separation of the 1,6- and 1,7-isomers.

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