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GAS-LIQUID CHROMATOGRAPHIC ANALYSES

XXXIV*. SEPARATION AND RETENTION INDICES WITH RETENTION INCREMENTS OF SOME NITRATED POLYNUCLEAR AROMATIC HYDROCARBONS ON A LOW-POLARITY (SE-30) CAPILLARY COLUMN

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SUMMARY

The gas chromatographic separation of a mixture of some polycyclic aromatic hydrocarbons (PAHs) and their nitro derivatives (nitro-PAHs) was studied on an SE-30 quartz capillary column under various temperature-programmed and isothermal operating conditions. The relative retention data and the Kováts retention indices, relative to *n*-alkanes, and the PAC retention indices, relative to polycyclic aromatic compounds, *viz.*, naphthalene, phenanthrene, chrysene and picene, together with both retention index increments due to the nitro substitution are given. The results are examined and compared with those reported previously for PAHs and nitro-PAHs.

INTRODUCTION

The importance of nitrated polynuclear aromatic hydrocarbons (nitro-PAHs) has been increased since 1978 when Pitts *et al.*¹ demonstrated that the PAHs react with nitrogen oxides to form nitro-PAHs. These components are of considerable interest, *e.g.*, as air and water pollutants, owing to their mutagenicity and/or carcinogenicity. Therefore, various techniques have been used and developed for the trace analysis of the nitro-PAHs in several environmental samples²⁻⁴.

The gas chromatographic (GC) retention times, the relative retention times or particularly the retention indices of the compounds can be used for the identification of the isomers, which otherwise are undifferentiated, *e.g.*, based on their identical mass spectral fragmentations. The Kováts retention indices for isothermal and temperature-programmed runs have been most frequently reported for PAHs⁵⁻¹², but are not available for the nitro-PAHs. Because of the problems encountered with the retention indices of PAHs using *n*-alkanes as internal standards, a linear retention index system for the temperature-programmed capillary GC of the polycyclic aro-

* For Part XXXIII, see *J. Chromatogr.*, 321 (1985) 467.

matic compounds (PACs) using naphthalene, phenanthrene, chrysene and picene as internal standards has been developed by Lee and co-workers^{13,14}; the retention indices of a wide range of aromatics are available, including some nitro-PAHs¹⁴. The effect on retention of the nitro substitution has not been reported, however.

This paper reports the separation of a mixture of some commercially available PAHs and nitro-PAHs on a low-polarity SE-30 quartz capillary column under various isothermal and temperature-programmed conditions. The relative retention data and the Kováts and PAC retention indices, together with the retention increments of both retention indices due to the nitro substitution, are examined and the results are compared with those reported previously for PAHs and nitro-PAHs⁵⁻¹⁴.

EXPERIMENTAL

Materials

Naphthalene (1), biphenyl (4), fluorene (8) and phenanthrene (11) were commercial products from Fluka (Buchs, Switzerland), chrysene (12) was obtained from Ega-Chemie (Steinheim, F.R.G.) and the nitro-PAHs (2, 3, 5-7, 9 and 10) from Foxboro/Analabs (North Haven, CT, U.S.A.). All samples were used without further purification.

Commercial mixtures of *n*-alkanes used as reference compounds were obtained from different sources.

Methods

GC analyses were carried out on a Perkin-Elmer Sigma 3 gas chromatograph under the following operating conditions: injection and flame-ionization detection temperatures, 300°C; nitrogen carrier gas velocity for methane at 180°C, 13 cm sec⁻¹; splitting ratio, 1:25; and chart speed, 10 mm min⁻¹. The column used was a vitreous silica SE-30 wall-coated open-tubular (WCOT) column (25 m × 0.33 mm I.D.), supplied by SGE (North Melbourne, Australia). The column temperature was programmed from 100 to 320°C at 10°C min⁻¹ and from 140 to 320°C at 6°C min⁻¹; the isothermal data were recorded at 180, 200, 220 and 240°C.

The retention times were measured from the time of sample injection, a Hewlett-Packard Model 3390A reporting integrator being used. The retention indices were calculated off-line by using two appropriate *n*-alkanes (Kováts retention indices¹⁵) or PAH internal standards (PAC retention indices^{13,14}), the dead volumes for the isothermal runs being determined by the injection of methane.

RESULTS AND DISCUSSION

Chromatograms of a mixture of the compounds analysed are shown in Figs. 1 and 2, separated with temperature programming and isothermal operation, respectively. The corresponding retention data, with various relative retention times, are presented in Table I.

The compounds were in general eluted in order of increasing boiling point, as usual on a low-polarity column. The mixture of twelve components analysed showed eleven resolved peaks (Fig. 1); 1-nitronaphthalene (2) was eluted as an unresolved shoulder with fluorene (8). Table I shows that the relative retention times with tem-

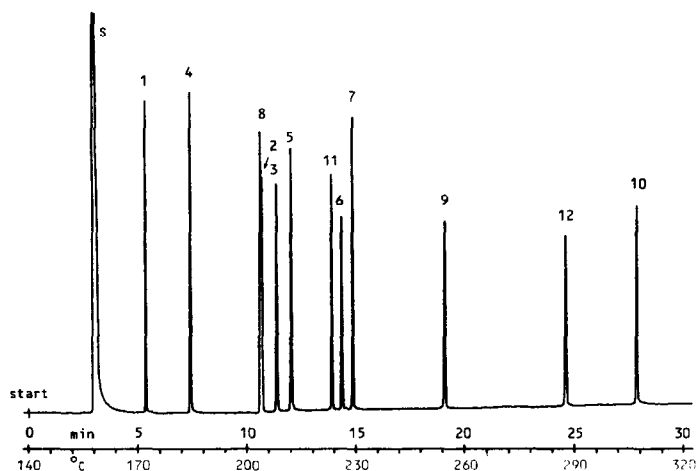


Fig. 1. Chromatogram of a mixture of the PAHs and nitro-PAHs, separated on an SE-30 quartz capillary column with temperature programming from 140 to 320°C at 6°C min⁻¹. S = Solvent; peaks are identified in Table I.

perature programming, relative to the parent components (1, 4 and 8), varied between 1.62 (2-nitrobiphenyl) and 2.14 (2-nitronaphthalene). With isothermal operation the variation was larger, *i.e.*, from 1.60 (2-nitrobiphenyl) to 3.03 (2-nitrofluorene).

The Kováts and PAC retention indices of the compounds studied are shown in Tables II and III, respectively, and are in accordance with those reported previously on several packed and capillary columns with low-polarity stationary phases, *viz.*, OV-19^{9,12}, OV-17^{5-7,11}, OV-101^{5,6,10}, SE-30^{9,11,12} and SE-52^{8,13,14}. With the nitro-PAHs, however, no comparisons could be made, because PAC retention indices on only one column (SE-52) with temperature programming have been reported¹⁴,

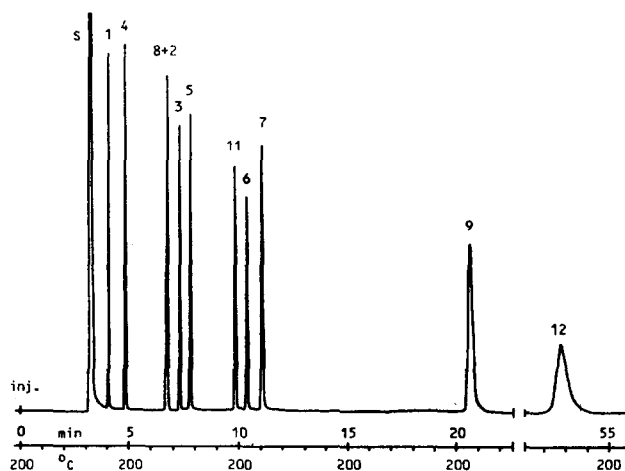


Fig. 2. Chromatogram of a mixture of the PAHs and nitro-PAHs, separated on an SE-30 quartz capillary column at 200°C. S = Solvent; peaks are identified in Table I. The peak of 4-nitro-*p*-terphenyl (10) is omitted owing to its long retention time (106.95 min).

TABLE I

RETENTION DATA FOR SOME PAHs AND THEIR NITRO DERIVATIVES, OBTAINED ON SE-30 WITH TEMPERATURE PROGRAMMING AND ISOTHERMAL CONDITIONS

Conditions as in Figs. 1 and 2.

Peak No.	Compound	Column (SE-30) temperature				
		Programmed from 140 to 320°C at 6°C min ⁻¹				
		ART*	RRT**	RRT***	RRT [§]	RRT ^{¶¶}
1	Naphthalene ^{§§§}	5.31	1.00	0.38	0.22	1.00
2	1-Nitronaphthalene	10.64	2.00	0.77	0.43	2.00
3	2-Nitronaphthalene	11.37	2.14	0.82	0.46	2.14
4	Biphenyl	7.40	1.39	0.53	0.30	1.00
5	2-Nitrobiphenyl	12.02	2.26	0.87	0.49	1.62
6	3-Nitrobiphenyl	14.32	2.70	1.03	0.58	1.94
7	4-Nitrobiphenyl	14.81	2.79	1.07	0.60	2.00
8	Fluorene	10.61	2.00	0.76	0.43	1.00
9	2-Nitrofluorene	19.09	3.60	1.38	0.78	1.80
10	<i>p</i> -Terphenyl [†]	—	—	—	—	—
	4-Nitro- <i>p</i> -terphenyl	27.86	5.25	2.01	1.13	—
11	Phenanthrene ^{§§§}	13.87	2.61	1.00	0.56	—
12	Chrysene ^{§§§}	24.62	4.64	1.78	1.00	—

* Absolute retention times (min) were measured from sample injection (Figs. 1 and 2).

** Relative retention time for naphthalene (1) taken as 1.00.

*** Relative retention time for phenanthrene (11) taken as 1.00.

§ Relative retention time for chrysene (12) taken as 1.00.

¶ Relative retention time for the corresponding parent compound (1, 4 and 8) taken as 1.00.

§§ Reference compounds.

† Compound not available.

†† From ref. 16.

††† From ref. 17.

the Kováts retention indices being not available. The disparities observed are due, e.g., to the different column and its length, the concentration of the stationary phase, the column temperature, the gas flow-rate and the injection system⁹. Other reasons reported are small differences in the operating conditions, such as an initial isothermal period¹⁴, and the difference in the sample matrices¹⁸.

The Kováts retention indices (Table II) increase with increasing operating temperature from 180 to 240°C, the variation ΔI per 20°C lying in the range 2–48 retention index units (i.u.). The variation between the retention indices obtained with temperature programming is lower, i.e., from -10 to 15 i.u. Unlike the Kováts retention indices, the PAC retention indices decrease with increasing column temperature, i.e., $\Delta I_{200^\circ\text{C}} - \Delta I_{180^\circ\text{C}}$ is in the range from -0.35 to -1.66 i.u., the difference $\Delta I_{220^\circ\text{C}} - \Delta I_{200^\circ\text{C}}$ being between -0.76 and -2.79 i.u. However, on increasing the column temperature from 220 to 240°C, the values increase from 0.37

<i>Isothermal at 200°C</i>					<i>B.p.</i> ^{††} (°C/mmHg)	<i>M.p.</i> ^{††} (°C)
<i>ART</i> [*]	<i>RRT</i> ^{**}	<i>RRT</i> ^{***}	<i>RRT</i> [§]	<i>RRT</i> [¶]		
4.09	1.00	0.41	0.08	1.00	218	80.5
6.80	1.66	0.69	0.13	1.66	304 (sublimes)	61.5
7.33	1.79	0.74	0.14	1.79	312.5/734	79
4.86	1.19	0.49	0.09	1.00	255.9	71
7.80	1.91	0.79	0.15	1.60	320	37.2
10.41	2.55	1.05	0.20	2.14	225-230/35	62
11.10	2.71	1.12	0.21	2.28	340	114
6.79	1.66	0.69	0.13	1.00	293-295	116-117
20.60	5.04	2.08	0.39	3.03	—	158
—	—	—	—	—	250/45 (sublimes)	213
106.95	26.15	10.81	2.02	—	—	214-215 ^{†††}
9.89	2.42	1.00	0.19	—	340	101
52.85	12.92	5.34	1.00	—	448	255-256

to 2.04 i.u., except for those of the last-eluted 2-nitrofluorene (9) and 4-nitro-*p*-terphenyl (10). Obviously this is due to the fact that the temperature used of 240°C is too high for the volatile components, particularly for naphthalene used as a reference component. With temperature programming the variation between the PAC retention indices is in the range from -1.58 to 7.50 i.u., considerably larger than with isothermal operation, as a consequence of the different initial temperature and the programming rate (Table III).

The Kováts and PAC retention increments due to the nitro substitution are shown in Tables IV and V, respectively. It is evident¹⁹ that the retention increment for nitrobenzene relative to benzene on low-polarity Apiezon L stationary phase is 406 i.u., in accordance with the values presented in Table IV. As shown, the retention increment is most frequently between 400 and 500 i.u. and generally increases with increasing temperature or programming rate. The lowest increments with the com-

TABLE II
 KOVÁTS RETENTION INDICES FOR PAHs AND NITRO-PAHs, DETERMINED ON SE-30 AT VARIOUS COLUMN TEMPERATURES
 Conditions as, e.g., in Figs. 1 and 2.

Peak No.	Compound	Column (SE-30) temperature		Isothermal															
		Programmed		140-320°C at 180°C				200°C				220°C				240°C			
		I^*	ΔI_1^{**}	I^*	ΔI_1^{**}	I^*	ΔI_2^{***}	I^*	ΔI_3^{\S}	I^*	ΔI_3^{\S}	I^*	ΔI_4^{\P}	I^*	ΔI_4^{\P}	I^*	ΔI_4^{\P}		
1	Naphthalene	1189	-10	1199		1210		1242		1244		1244		1262		1262			
2	1-Nitronaphthalene	1586	-3	1589		1597		1618		1625		1625		1662		1662			
3	2-Nitronaphthalene	1631	0	1631		1635		1656		1672		1672		1708		1708			
4	Biphenyl	1374	-4	1378		1387		1408		1411		1411		1433		1433			
5	2-Nitrobiphenyl	1669	0	1669		1670		1686		1698		1698		1736		1736			
6	3-Nitrobiphenyl	1808	6	1802		1792		1813		1831		1831		1865		1865			
7	4-Nitrobiphenyl	1834	2	1832		1816		1839		1856		1856		1891		1891			
8	Fluorene	1583	-4	1587		1595		1617		1625		1625		1662		1662			
9	2-Nitrofluorene	2102	15	2087		2037		2066		2095		2095		2129		2129			
10	<i>p</i> -Terphenyl	2208 ^{§§§}	-	2208 ^{§§§}		-		-		-		-		-		-			
	4-Nitro- <i>p</i> -terphenyl	2696	1	2695		-		2586		2620		2620		2654		2654			
11	Phenanthrene	1791	15	1776		1765		1792		1822		1822		1850		1850			
12	Chrysene	2465	11	2454		-		2371		2408		2408		2456		2456			

* Kováts retention index, relative to *n*-alkanes.

** ΔI_1 = Difference between the temperature-programmed runs.

*** $\Delta I_2 = I_{200^\circ\text{C}} - I_{180^\circ\text{C}}$

§ $\Delta I_3 = I_{220^\circ\text{C}} - I_{200^\circ\text{C}}$

¶ $\Delta I_4 = I_{240^\circ\text{C}} - I_{220^\circ\text{C}}$

§§ From ref. 9, on packed columns coated with low-polarity SE-30 or OV-1.

TABLE III
 PAC RETENTION INDICES FOR PAHs AND NITRO-PAHs, DETERMINED ON SE-30 AT VARIOUS COLUMN TEMPERATURES
 Conditions as, e.g., in Figs. 1 and 2.

Peak No.	Compound	Column (SE-30) temperature											
		Programmed						Isothermal					
		100-320°C at 10°C min ⁻¹		140-320°C at 6°C min ⁻¹		180°C		200°C		220°C		240°C	
<i>t</i> [*]	ΔI_1^{**}	<i>t</i> [*]	<i>t</i> [*]	<i>t</i> [*]	ΔI_2^{***}	<i>t</i> [*]	ΔI_3^{\S}	<i>t</i> [*]	ΔI_4^{\P}	<i>t</i> [*]	ΔI_4^{\P}	<i>t</i> [*]	
1	Naphthalene	200.00	—	200.00	—	200.00	—	200.00	—	200.00	—	—	200.00
2	1-Nitronaphthalene	267.95	5.68	262.27	—	269.78	-2.63	269.20	-2.63	266.57	1.43	—	268.00
3	2-Nitronaphthalene	275.23	4.44	270.79	—	276.73	-1.63	276.02	-1.63	274.39	1.64	—	276.03
4	Biphenyl	231.92	7.50	224.42	—	232.03	-2.28	230.79	-2.28	228.51	0.37	—	228.88
5	2-Nitrobiphenyl	281.46	3.07	278.39	—	283.04	-1.66	281.38	-1.66	278.59	2.04	—	280.63
6	3-Nitrobiphenyl	302.72	-1.47	304.19	—	304.82	-1.09	303.73	-2.30	301.43	1.16	—	302.59
7	4-Nitrobiphenyl	307.16	-1.58	308.74	—	308.99	-0.70	308.29	-2.55	305.74	1.13	—	306.87
8	Fluorene	267.42	5.50	261.92	—	269.55	-0.49	269.06	-2.49	266.57	1.43	—	268.00
9	2-Nitrofluorene	348.28	-0.28	348.56	—	348.02	-0.35	347.67	-0.76	346.91	-0.10	—	346.81
10	<i>p</i> -Terphenyl	366.10 ^{¶¶}	—	366.10 ^{¶¶}	—	—	—	—	—	—	—	—	—
	4-Nitro- <i>p</i> -terphenyl	437.54	7.40	430.14	—	—	—	436.79	-1.23	435.56	-3.80	—	431.76
11	Phenanthrene	300.00	—	300.00	—	300.00	—	300.00	—	300.00	—	—	300.00
12	Chrysene	400.00	—	400.00	—	400.00	—	400.00	—	400.00	—	—	400.00

* PAC retention index, relative to naphthalene (= 200.00), phenanthrene (= 300.00) and chrysene (= 400.00)^{13,14}.

** ΔI_1 = Difference between the temperature-programmed runs.

*** $\Delta I_2 = I_{200^\circ\text{C}} - I_{180^\circ\text{C}}$.

§ $\Delta I_3 = I_{220^\circ\text{C}} - I_{200^\circ\text{C}}$.

¶ $\Delta I_4 = I_{240^\circ\text{C}} - I_{220^\circ\text{C}}$.

¶¶ From ref. 13, on a capillary column coated with low-polarity SE-52.

TABLE IV
KOVÁTS RETENTION INCREMENTS FOR NITRO-PAHs AT VARIOUS COLUMN TEMPERATURES

Compound	Column (SE-30) temperature													
	Programmed							Isothermal						
	100-320°C at 10°C min ⁻¹		140-320°C at 180°C 6°C min ⁻¹					200°C		220°C		240°C		
ΔI^*	ΔI_1^{**}	ΔI^*	ΔI_2^{***}	ΔI^*	ΔI_3^{\S}	ΔI^*	ΔI^*	$\Delta I_4^{\S\S}$	ΔI^*	ΔI^*	ΔI_3^{\S}	ΔI^*	$\Delta I_4^{\S\S}$	ΔI^*
1-Nitronaphthalene	397	7	390	387	387	376	376	381	381	376	5	381	19	400
2-Nitronaphthalene	442	10	432	425	425	414	414	428	428	414	14	428	18	446
2-Nitrobiphenyl	295	4	291	283	283	278	278	287	287	278	9	287	16	303
3-Nitrobiphenyl	434	10	424	405	405	405	405	420	420	405	15	420	12	432
4-Nitrobiphenyl	460	6	454	429	429	431	431	445	445	431	14	445	13	458
2-Nitrofluorene	519	19	500	442	442	449	449	470	470	449	21	470	-3	467
4-Nitro- <i>p</i> -terphenyl	488 ^{§§§}	1	487 ^{§§§}	-	-	-	-	-	-	-	-	-	-	-

* For the Kováts retention indices of the compounds, see Table II.

** ΔI_1 = Difference between the programmed runs.

*** ΔI_2 = $\Delta I_{200^\circ\text{C}} - \Delta I_{180^\circ\text{C}}$.

§ ΔI_3 = $\Delta I_{220^\circ\text{C}} - \Delta I_{200^\circ\text{C}}$.

§§ ΔI_4 = $\Delta I_{240^\circ\text{C}} - \Delta I_{220^\circ\text{C}}$.

§§§ Approximate value.

TABLE V
PAC RETENTION INCREMENTS FOR NITRO-PAHs AT VARIOUS COLUMN TEMPERATURES

Compound	Column temperature										
	Programmed					Isothermal					
	100-320°C at 10°C min ⁻¹		140-320°C at 6°C min ⁻¹		40-265°C at 4°C min ⁻¹ ***	180°C	200°C	220°C	240°C		
	ΔI^*	ΔI_1^{***}	ΔI^*	ΔI^*	ΔI^*	ΔI_2^{\S}	ΔI^*	ΔI_3^{\P}	ΔI^*	$\Delta I_4^{\text{¶}}$	ΔI^*
1-Nitronaphthalene	67.95	5.58	62.27	74.95	69.78	-0.58	69.20	-2.63	66.57	1.43	68.00
2-Nitronaphthalene	75.23	4.44	70.79	80.63	76.73	-0.71	76.02	-1.63	74.39	1.64	76.03
2-Nitrobiphenyl	49.54	-4.43	53.97	53.81	51.01	-0.42	50.59	-0.51	50.08	1.67	51.75
3-Nitrobiphenyl	70.80	-8.97	79.77	73.65	72.79	0.15	72.94	-0.02	72.92	0.79	73.71
4-Nitrobiphenyl	75.24	-9.08	84.32	78.15	76.96	0.54	77.50	-0.27	77.23	0.76	77.99
2-Nitrofluorene	80.86	-5.78	86.64	83.33	78.47	0.14	78.61	1.73	80.34	-1.53	78.81
9-Nitroanthracene	-	-	-	56.34	-	-	-	-	-	-	-
1-Nitropyrene	-	-	-	69.97	-	-	-	-	-	-	-
4-Nitro- <i>p</i> -terphenyl	71.44†	7.40	64.04†	-	-	-	-	-	-	-	-
6-Nitrobenzo(a)pyrene	-	-	-	47.69	-	-	-	-	-	-	-

* For the PAC retention indices of the compounds, see Table III.

** From ref. 14, on low-polarity SE-52 fused silica columns with a 2-min initial isothermal period.

*** ΔI_1 = Difference between the temperature-programmed runs.

§ ΔI_2 = $\Delta I_{200^\circ\text{C}} - \Delta I_{180^\circ\text{C}}$.

¶ ΔI_3 = $\Delta I_{220^\circ\text{C}} - \Delta I_{200^\circ\text{C}}$.

‡ ΔI_4 = $\Delta I_{240^\circ\text{C}} - \Delta I_{220^\circ\text{C}}$.

† Approximate value.

ponents studied are shown by 2-nitrobiphenyl, *i.e.*, 283–303 i.u. at various temperatures; the values increase to 405–434 i.u. with 3-nitrobiphenyl and to 429–460 i.u. with 4-nitrobiphenyl. The disparities between these three isomers observed at 180°C are 122 and 24 i.u., respectively. As is evident from the series benzene (406 i.u.) → biphenyl (460 i.u.) → *p*-terphenyl (488 i.u.), the increment due to *p*-nitro substitution given in parentheses increases with increasing size of the parent compound. The same effect is shown with phenyl substitution in the series benzene (697 i.u.¹⁹) → biphenyl (1389 i.u.⁹) → *p*-terphenyl (2208 i.u.⁹), the retention indices of the components being given in parentheses, *i.e.*, the retention increment of the first phenyl substituent is 692 i.u. and that of the second phenyl substituent is 819 i.u. The series nitrobenzene (1103 i.u.¹⁹) → 4-nitrobiphenyl (1834, Table II) → 4-nitro-*p*-terphenyl (2208 i.u.⁹) shows higher increments for phenyl substitution, *viz.*, 731 and 862 i.u., respectively.

Generally, the same observations as mentioned above can be seen from the data presented in Table V, although the increases in the PAC retention index increments with increasing temperature are not so evident. The data presented, which include the values for three additional compounds calculated from the retention indices of Vassilaros *et al.*¹⁴, show that unlike the Kováts retention increments the PAC retention increments decrease with increasing size, *i.e.*, the molecular weight of the parent component. However, as with naphthalene and biphenyl, the position of substitution may affect the retention behaviour also with the other compounds studied, shown, *e.g.*, by a lower increment for 9-nitroanthracene with respect to 1-nitropyrene (Table V), but this is uncertain as it is based on the GC analysis of only one isomer, carried out previously¹⁴ and in this work.

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REFERENCES

- 1 J. N. Pitts, Jr., K. A. van Cauwenberghe, D. Grosjean, J. T. Schmid, D. R. Fitz, W. L. Belser, Jr., G. B. Knudson and P. M. Hynds, *Science*, 202 (1978) 515.
- 2 T. Ramdahl and K. Urdal, *Anal. Chem.*, 54 (1982) 2256, and references cited therein.
- 3 M. C. Paputa-Peck, R. S. Marano, D. Schuetzle, T. L. Riley, C. V. Hampton, T. J. Prater, L. M. Skewes, T. E. Jensen, P. H. Ruehle, L. C. Bosch and W. P. Duncan, *Anal. Chem.*, 55 (1983) 1946, and references cited therein.
- 4 I. S. Krull, M. Swartz, R. Hilliard, K.-H. Xie and J. N. Driscoll, *J. Chromatogr.*, 260 (1983) 347, and references cited therein.
- 5 G. Grimmer and H. Böhnke, *Z. Anal. Chem.*, 261 (1972) 310.
- 6 R. Kaliszan and H. Lamparczyk, *J. Chromatogr. Sci.*, 16 (1978) 246.
- 7 I. M. Mutton, *J. Chromatogr.*, 172 (1979) 438.
- 8 H. Beernaert, *J. Chromatogr.*, 173 (1979) 109.
- 9 J. D. Ramsey, T. D. Lee, M. D. Osselson and A. C. Moffat, *J. Chromatogr.*, 184 (1980) 185.
- 10 V. A. Gerasimenko, A. V. Kirilenko and V. M. Nabivach, *J. Chromatogr.*, 208 (1981) 9, and references cited therein.
- 11 F. Sellier, G. Tersac and G. Guiochon, *J. Chromatogr.*, 219 (1981) 213.
- 12 R. E. Ardrey and A. C. Moffat, *J. Chromatogr.*, 220 (1981) 195.
- 13 M. L. Lee, D. L. Vassilaros, C. M. White and M. Novotny, *Anal. Chem.*, 51 (1979) 768.

- 14 D. L. Vassilaros, R. C. Kong, D. W. Later and M. L. Lee, *J. Chromatogr.*, 252 (1982) 1.
- 15 G. Guiochon, *Anal. Chem.*, 36 (1964) 661.
- 16 R. C. Weast (Editor), *CRC Handbook of Chemistry and Physics*, CRC Press, Boca Raton, FL, 62nd ed., 1981.
- 17 W. S. M. Grieve and D. H. Hey, *J. Chem. Soc.*, (1938) 108.
- 18 H. Y. Tong, D. L. Shore, F. W. Karasek, P. Helland and E. Jellum, *J. Chromatogr.*, 285 (1984) 423.
- 19 F. Vernon, *J. Chromatogr.*, 87 (1973) 29.