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GAS-LIQUID CHROMATOGRAPHIC ANALYSES

XLVI*. SEPARATION OF C₃–C₅ BRANCHED-CHAIN, C₃–C₆ UNSATURATED AND ETHYL AND ω -CHLOROETHYL ESTERS OF BENZOIC, 4-NITROBENZOIC AND 3,5-DINITROBENZOIC ACIDS ON SE-30 AND OV-351 CAPILLARY COLUMNS WITH TEMPERATURE PROGRAMMING

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SUMMARY

The separations of the mixtures of (i) six lower (C₃–C₅) saturated branched-chain, (ii) eight lower (C₃–C₆) unsaturated and (iii) ethyl, 2-chloroethyl, 2,2-dichloroethyl and 2,2,2-trichloroethyl esters of benzoic, 4-nitrobenzoic and 3,5-dinitrobenzoic acids on low-polarity (SE-30) and highly polar (OV-351) quartz capillary columns with temperature programming were investigated. The retention data for all 54 components are given and their elution orders on both columns are discussed, together with the effects of nitro substitution in the acyl chain and chlorine substitution in the alkyl chain.

INTRODUCTION

Our previous gas chromatographic (GC) studies with lower esters involved the separation and retention behaviour of saturated branched-chain^{1–6}, unsaturated^{3,4,6–9} and ethyl and ω -chloroethyl^{10–12} esters of acetic, chloroacetic, dichloroacetic, trichloroacetic, bromoacetic, dibromoacetic and tribromoacetic acids^{1,7}, propanoic, 2- and 3-chloropropanoic acids^{2,3,8}, butanoic, 2-, 3- and 4-chlorobutanoic acids^{2,4,8}, benzoic, 2-, 3- and 4-chlorobenzoic acids^{5,9,10}, pentafluorobenzoic acid⁶ and aliphatic C₂–C₂₀ *n*-alkanoic acids^{11,12}. To optimize the separations of the individual components in the complex mixtures, both low-polarity (SE-30) and highly polar (OV-351) capillary columns with suitable temperature programming were used^{1,2,5–12}. The effects of chain branching, unsaturation and chlorine substitution, shown by the retention index increments, were most frequently examined based on isothermal data obtained at a variety of temperatures^{3–6,9,10,12}.

The GC of nitrobenzoic acid esters has not been widely investigated, as reported in the last paper dealing with the retention behaviour of C₁–C₁₂ *n*-alkyl esters

* For Part XLV, see ref. 13.

of 4-nitro- and 3,5-dinitrobenzoic acids on SE-30 and OV-351 capillary columns^{1,3}. This paper describes the GC of six saturated branched-chain, eight unsaturated and ethyl and ω -chloroethyl esters of benzoic, 4-nitrobenzoic and 3,5-dinitrobenzoic acids. The mixtures of these (i) 18, (ii) 24 and (iii) 12 esters were separated on low-polarity (SE-30) and highly polar (OV-351) quartz capillary columns with temperature programming. The absolute and relative retention data for all 54 compounds are given and the retention behaviour on both columns is discussed, together with the effects of nitro and chloro substitutions. The results are compared with those reported previously for the corresponding esters of halobenzoic acids^{5,6,9,10}.

EXPERIMENTAL

Materials

Saturated branched-chain, unsaturated and ethyl and ω -chloroethyl esters of benzoic (1-18), 4-nitrobenzoic (4/1-4/18) and 3,5-dinitrobenzoic (35/1-35/18) acids were synthesized from the corresponding branched-chain alcohols (Fluka, Buchs, Switzerland), unsaturated alcohols (Fluka or Merck-Schuchardt, Darmstadt, F.R.G.), ethanol (Oy Alko, Finland), 2-chloroethanol (Fluka), 2,2-dichloroethanol¹⁴ and 2,2,2-trichloroethanol¹⁴ with commercial (Merck-Schuchardt) benzoyl, 4-nitrobenzoyl and 3,5-dinitrobenzoyl chlorides as described earlier¹⁵. The esters investigated are listed in Tables I-III.

n-Tetradecane used as a reference component was obtained from Fluka.

Methods

GC analyses were carried out on a Perkin-Elmer Sigma 3 instrument under the following operating conditions: injection and flame-ionization detection (FID) temperatures, 260°C; nitrogen carrier gas velocities for methane, 27.2 (SE-30) and 59.5 cm s⁻¹ (OV-351); splitting ratio, 1:30; and chart speed, 10 mm min⁻¹. The columns used were a vitreous silica SE-30 wall-coated open-tubular (WCOT) column (25 m × 0.33 mm I.D.), supplied by SGE (North Melbourne, Australia), and a fused-silica OV-351 WCOT column (25 m × 0.32 mm I.D.), supplied by Orion Analytica (Espoo, Finland). The column temperature was programmed from 100°C at 6°C min⁻¹ until elution of peaks had ceased (SE-30) and from 100 to 220°C at 6°C min⁻¹ and held at 220°C until elution of peaks had ceased (OV-351).

The chromatographic data were recorded with a Hewlett-Packard Model 3390A reporting integrator and the retention times were measured from the time of sample injection.

The three model mixtures analysed contained suitable amounts of the individual pure components for the sensitivity of the flame-ionization detector and the response of the column. The order of elution of the compounds was confirmed by using a standard additions method.

RESULTS AND DISCUSSION

Chromatograms of mixtures of (i) saturated branched-chain, (ii) unsaturated and (iii) ethyl and ω -chloroethyl esters of benzoic, 4-nitrobenzoic and 3,5-dinitrobenzoic acids are illustrated in Figs. 1-6, obtained on SE-30 and OV-351 capillary

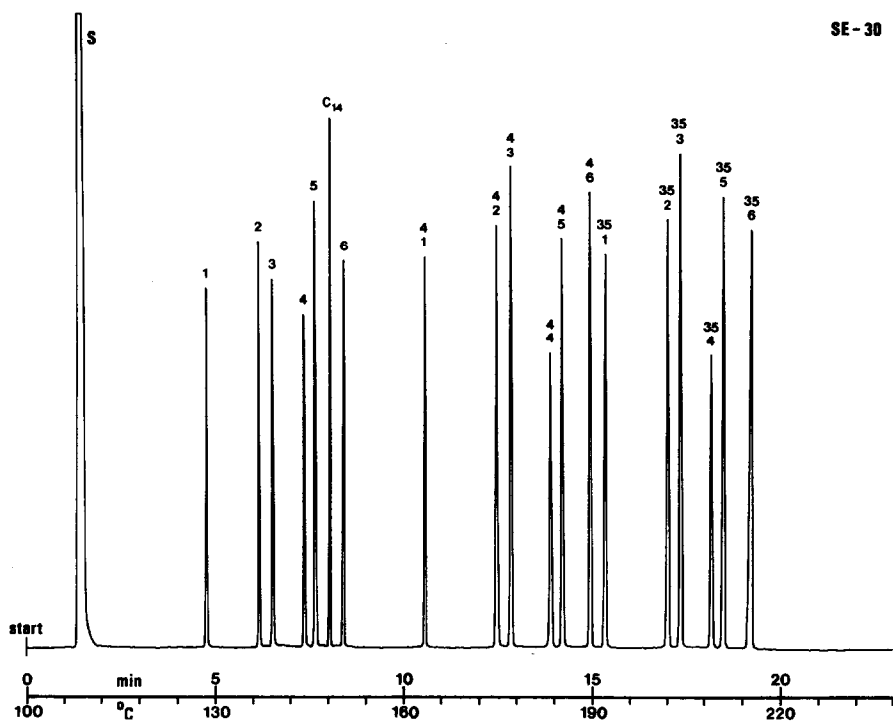


Fig. 1. Chromatogram of a mixture of saturated branched-chain esters of benzoic (1-6), 4-nitrobenzoic (4/1-4/6) and 3,5-dinitrobenzoic (35/1-35/6) acids, separated on an SE-30 quartz capillary column with temperature programming from 100°C at 6°C min⁻¹ until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table I.

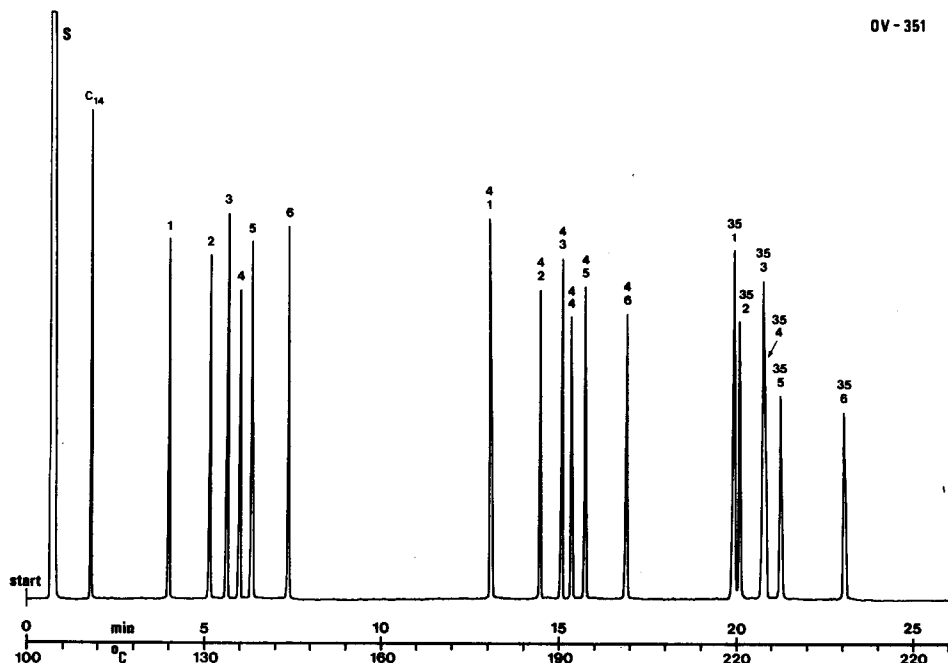


Fig. 2. Chromatogram of the same mixture as in Fig. 1, obtained on an OV-351 quartz capillary column with temperature programming from 100 to 220°C at 6°C min⁻¹ and held at 220°C until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table I.

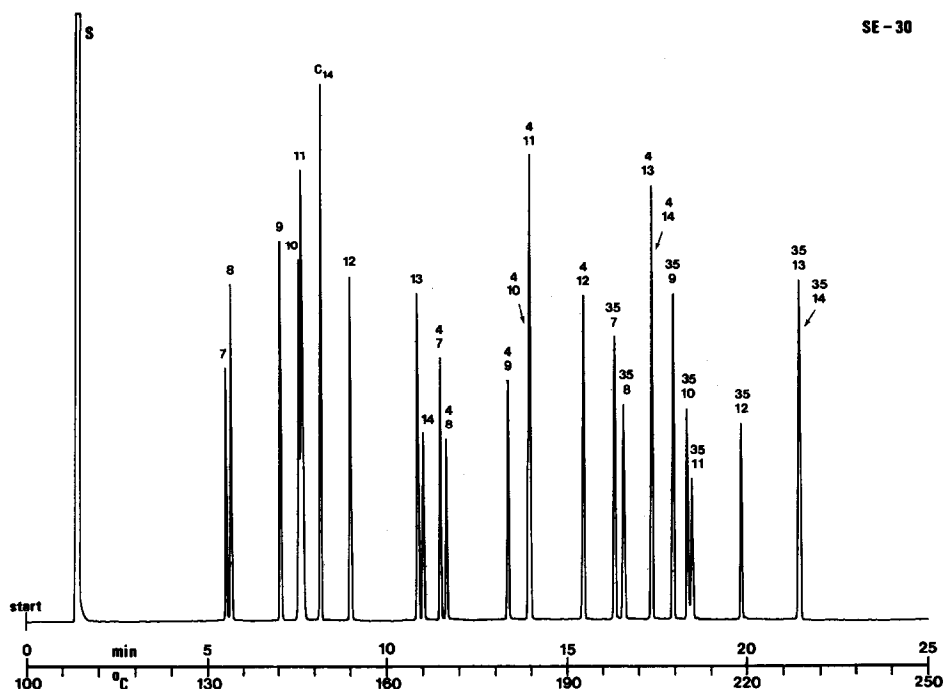


Fig. 3. Chromatogram of a mixture of unsaturated esters of benzoic (7-14), 4-nitrobenzoic (4/7-4/14) and 3,5-dinitrobenzoic (35/7-35/14) acids, separated on SE-30 with temperature programming from 100°C at 6°C min⁻¹ until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table II.

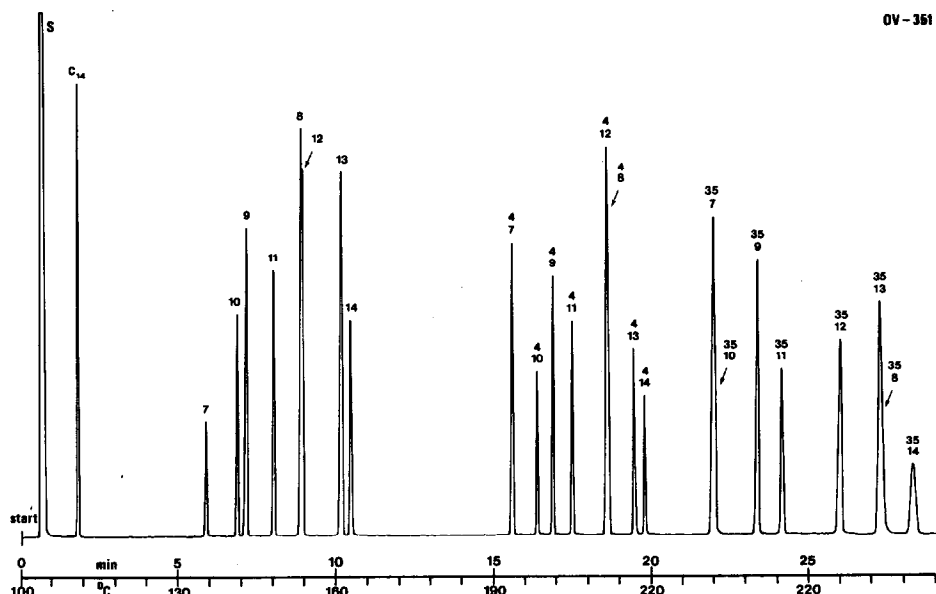


Fig. 4. Chromatogram of the same mixture as in Fig. 3, obtained on OV-351 with temperature programming from 100 to 220°C at 6°C min⁻¹ and held at 220°C until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table II.

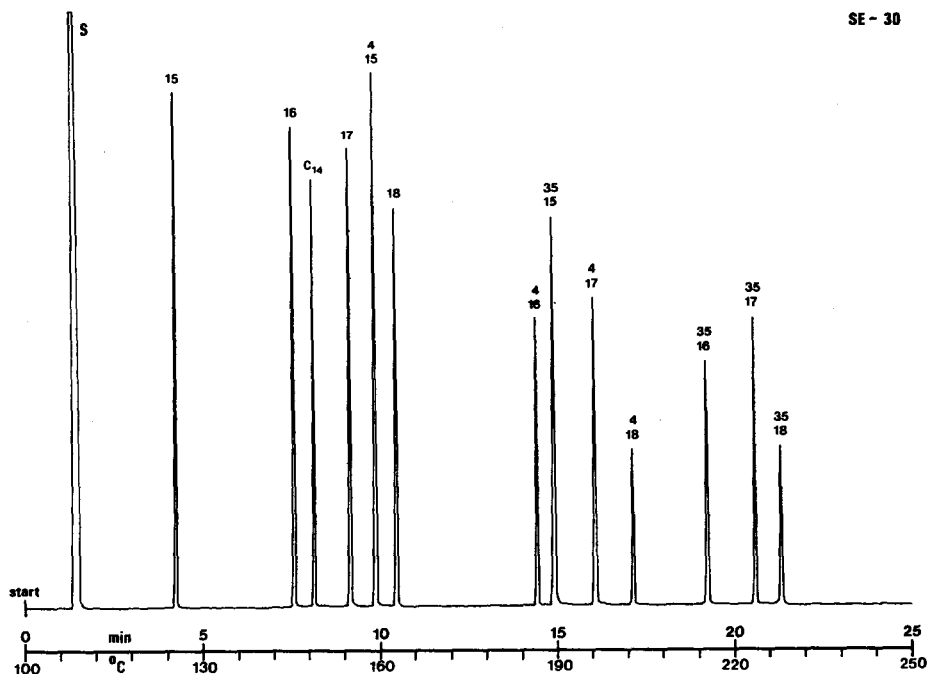


Fig. 5. Chromatogram of a mixture of ethyl and ω -chloroethyl esters of benzoic (15–18), 4-nitrobenzoic (4/15–4/18) and 3,5-dinitrobenzoic (35/15–35/18) acids, separated on SE-30 with temperature programming from 100°C at 6°C min⁻¹ until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table III.

columns with temperature programming. The absolute and relative retention data are presented in Tables I–III, a summary being shown in Table IV.

The separation of the branched-chain esters was complete on SE-30 (Fig. 1), whereas on OV-351 one compound pair is overlapped (Fig. 2). As would be expected, the nitro derivatives are eluted on both columns in the same order as their parent esters, although their mutual separation is changed, particularly with the 3,5-dinitrobenzoates on OV-351 (Fig. 2). Bearing in mind the retention behaviour of the corresponding *n*-alkyl esters¹³, saturated lower esters of all three groups on SE-30 follow the order methyl < ethyl < 1 < propyl < 2 < 3 < butyl < 4 < 5 < 6 < pentyl ester, the order being the same as reported earlier for 4-nitrobenzoates¹⁶ and 3,5-dinitrobenzoates¹⁷ on packed columns coated with low-polarity OV-17 and/or SE-30 stationary phases. Correspondingly, on OV-351 benzoates and 4-nitrobenzoates follow the order methyl < 1 ≅ ethyl < propyl < 2 < 3 < 4 < 5 < butyl < 6 < pentyl ester and 3,5-dinitrobenzoates the order methyl < ethyl < 1 < 2 < propyl < 3 < 4 < 5 < butyl < 6 < pentyl ester. Generally, the same orders are found as previously for the various groups of aliphatic^{1–4} and aromatic^{5,6} esters, the structure of the acyl chain having no effect on the retention order of the compounds on SE-30 and also a negligible effect on OV-351, where such effects might be maximal.

As with the branched-chain esters, the nitro substitution does not give rise to changes in the elution order of the unsaturated esters on SE-30, but leads to some

overlapping of the peaks (Fig. 3). The best resolution between the three 1-methyl-3-butenyl (10) and (*E*)-2-butenyl (11) esters is shown by the 3,5-dinitrobenzoates; benzoates are partially resolved, but 4-nitrobenzoates overlap. The (*E*)- and (*Z*)-3-hexenyl 4- and 3,5-derivatives are unresolvable on SE-30, as also are the esters of pentafluorobenzoic acid⁶. The corresponding monochlorobenzoate esters can be separated on SE-30, however⁹.

The reversed elution orders of the unsaturated esters on a highly polar OV-351 column (Fig. 4) generally are as found previously for the esters of halobenzoic acids^{6,9}. Excluding a single acetylenic ester (8 series), the retention order remains unchanged with all three groups of esters investigated. 2-Propynyl benzoate (8) is eluted close together with 4-pentenyl benzoate (12) and the mutual retention behaviour between the 4-nitro esters (4/8 and 4/12) remains nearly unchanged, although the elution order is changed (Fig. 4). With the 3,5-dinitro esters, however, the retention of the acetylenic ester (35/8) is markedly increased compared with that of the 4-pentenyl ester (35/12), 35/8 being even eluted later than the (*E*)-3-hexenyl isomer (35/13). This effect is the same, but much more pronounced, as found previously for the acetylenic pentafluorobenzoate⁶ and 2-chlorobenzoate⁹, not occurring with 3- and 4-chlorobenzoates, however⁹. The retention of a single branched-chain unsaturated ester (10) increases with the nitro substitution less than that of the *n*-esters, the 3,5-dinitro ester (35/10) eluting close together with the 2-propenyl ester (35/7), as is evident in Fig. 4. Like the monochlorinated (*E*)- and (*Z*)-3-hexenyl esters, the cor-

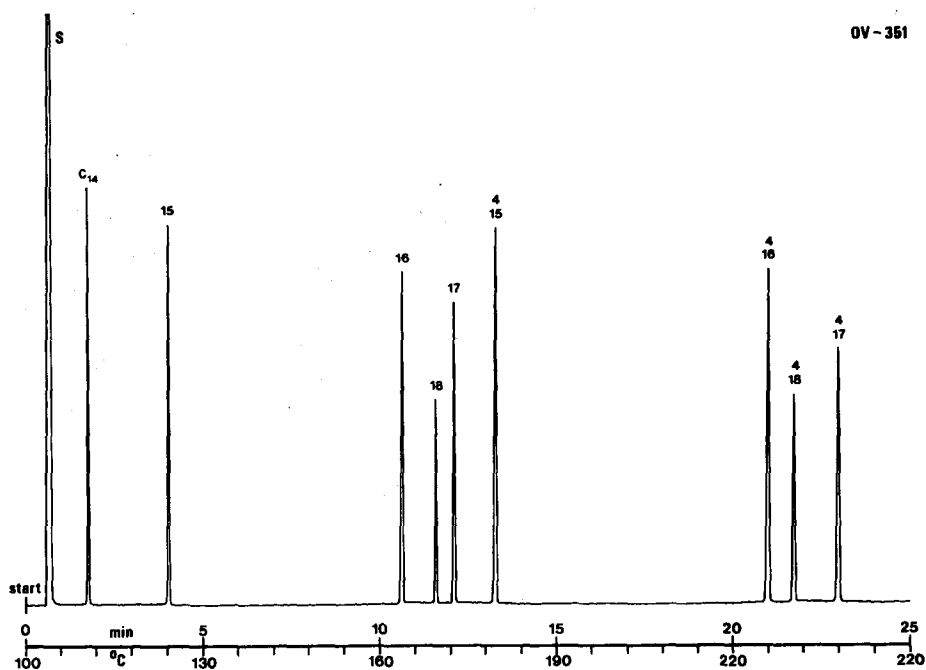


Fig. 6. Chromatogram of the same mixture as in Fig. 5, excluding the 3,5-dinitro esters, obtained on OV-351 with temperature programming from 100 to 220°C at 6°C min⁻¹ and held at 220°C until elution of peaks had ceased. S = Solvent; C₁₄ = *n*-tetradecane; peaks are listed in Table III.

TABLE I

RETENTION DATA FOR SATURATED BRANCHED-CHAIN ESTERS OF BENZOIC, 4-NITROBENZOIC AND 3,5-DINITROBENZOIC ACIDS, DETERMINED ON SE-30 AND OV-351 CAPILLARY COLUMNS WITH TEMPERATURE PROGRAMMING

Conditions as in Figs. 1 and 2.

Peak No.	Ester	Column						
		SE-30			OV-351			
		ART*	RRT**	RRT***	ART*	RRT**	RRT***	RRT§
<i>Benzoate</i>								
1	Methylethyl	4.75	0.59	1.00	4.00	2.25	1.00	0.84
2	1-Methylpropyl	6.16	0.76	1.00	5.17	2.90	1.00	0.84
3	2-Methylpropyl	6.51	0.80	1.00	5.67	3.19	1.00	0.87
4	1,2-Dimethylpropyl	7.37	0.91	1.00	6.01	3.38	1.00	0.82
5	1-Methylbutyl	7.61	0.94	1.00	6.37	3.58	1.00	0.84
6	3-Methylbutyl	8.39	1.04	1.00	7.38	4.15	1.00	0.88
<i>4-Nitrobenzoate</i>								
4/1	Methylethyl	10.54	1.30	2.22	13.08	7.35	3.27	1.24
4/2	1-Methylpropyl	12.43	1.53	2.02	14.49	8.14	2.80	1.17
4/3	2-Methylpropyl	12.81	1.58	1.97	15.11	8.49	2.66	1.18
4/4	1,2-Dimethylpropyl	13.89	1.71	1.88	15.38	8.64	2.56	1.11
4/5	1-Methylbutyl	14.19	1.75	1.86	15.73	8.84	2.47	1.11
4/6	3-Methylbutyl	14.92	1.84	1.78	16.91	9.50	2.29	1.13
<i>3,5-Dinitrobenzoate</i>								
35/1	Methylethyl	15.31	1.89	3.22	19.97	11.22	4.99	1.30
35/2	1-Methylpropyl	16.99	2.10	2.76	20.12	11.30	3.89	1.18
35/3	2-Methylpropyl	17.33	2.14	2.66	20.78	11.67	3.66	1.20
35/4	1,2-Dimethylpropyl	18.17	2.24	2.47	20.81	11.69	3.46	1.15
35/5	1-Methylbutyl	18.48	2.28	2.43	21.26	11.94	3.34	1.15
35/6	3-Methylbutyl	19.21	2.37	2.29	23.08	12.97	3.13	1.20
C ₁₄	<i>n</i> -Tetradecane	8.10	1.00	—	1.78	1.00	—	0.22

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

** Relative retention time for *n*-tetradecane (C₁₄) taken as 1.00.

*** Relative retention time for the corresponding ester of benzoic acid (1–6) taken as 1.00.

§ Relative retention time for the corresponding compound on SE-30 taken as 1.00.

responding nitro esters are resolvable on OV-351, whereas the pentafluoro esters showed only partially resolved peaks⁶.

The separation of ethyl and ω -chloroethyl esters, shown in Figs. 5 (SE-30) and 6 (OV-351), was complete on both columns. The 3,5-dinitro esters, eluting from SE-30, gave no peaks on OV-351, however. Obviously this is due to a too polar column for the chloro esters analysed and also to the instability of the esters, observed from their markedly decreased response on SE-30 compared with that of the corresponding 4-nitro isomers. The retention behaviour is as shown with the other es-

TABLE II

RETENTION DATA FOR UNSATURATED ESTERS OF BENZOIC, 4-NITROBENZOIC AND 3,5-DINITROBENZOIC ACIDS, DETERMINED ON SE-30 AND OV-351 CAPILLARY COLUMNS WITH TEMPERATURE PROGRAMMING

Conditions as in Figs. 3 and 4.

Peak No.	Ester	Column						
		SE-30			OV-351			
		ART*	RRT**	RRT***	ART*	RRT**	RRT***	RRT§
<i>Benzoate</i>								
7	2-Propenyl	5.51	0.68	1.00	5.87	3.30	1.00	1.07
8	2-Propynyl	5.65	0.70	1.00	8.82	4.96	1.00	1.56
9	3-Butenyl	6.99	0.86	1.00	7.16	4.02	1.00	1.02
10	1-Methyl-3-butenyl	7.46	0.92	1.00	6.86	3.85	1.00	0.92
11	(<i>E</i>)-2-Butenyl	7.51	0.93	1.00	8.02	4.51	1.00	1.07
12	4-Pentenyl	8.96	1.11	1.00	8.90	5.00	1.00	0.99
13	(<i>E</i>)-3-Hexenyl	10.79	1.33	1.00	10.17	5.71	1.00	0.94
14	(<i>Z</i>)-3-Hexenyl	10.99	1.36	1.00	10.46	5.88	1.00	0.95
<i>4-Nitrobenzoate</i>								
4/7	2-Propenyl	11.48	1.42	2.08	15.58	8.75	2.65	1.36
4/8	2-Propynyl	11.65	1.44	2.06	18.58	10.44	2.11	1.59
4/9	3-Butenyl	13.35	1.65	1.91	16.90	9.49	2.36	1.27
4/10	1-Methyl-3-butenyl	13.90	1.72	1.86	16.37	9.20	2.39	1.18
4/11	(<i>E</i>)-2-Butenyl	13.92	1.72	1.85	17.46	9.81	2.18	1.25
4/12	4-Pentenyl	15.42	1.90	1.72	18.50	10.39	2.08	1.20
4/13	(<i>E</i>)-3-Hexenyl	17.34	2.14	1.61	19.41	10.90	1.91	1.12
4/14	(<i>Z</i>)-3-Hexenyl	17.36	2.14	1.58	19.80	11.12	1.89	1.14
<i>3,5-Dinitrobenzoate</i>								
35/7	2-Propenyl	16.30	2.01	2.96	21.95	12.33	3.74	1.35
35/8	2-Propynyl	16.55	2.04	2.93	27.30	15.34	3.10	1.65
35/9	3-Butenyl	17.93	2.21	2.57	23.34	13.11	3.26	1.30
35/10	1-Methyl-3-butenyl	18.32	2.26	2.46	22.10	12.42	3.22	1.21
35/11	(<i>E</i>)-2-Butenyl	18.46	2.28	2.46	24.13	13.56	3.01	1.31
35/12	4-Pentenyl	19.81	2.45	2.21	26.00	14.61	2.92	1.31
35/13	(<i>E</i>)-3-Hexenyl	21.41	2.64	1.98	27.20	15.28	2.67	1.27
35/14	(<i>Z</i>)-3-Hexenyl	21.44	2.65	1.95	28.28	15.89	2.70	1.32
C ₁₄	<i>n</i> -Tetradecane	8.10	1.00	—	1.78	1.00	—	0.22

* Absolute retention times (min) were measured from the time of sample injection (Figs. 3 and 4).

** Relative retention time for *n*-tetradecane (C₁₄) taken as 1.00.

*** Relative retention time for the corresponding ester of benzoic acid (7–14) taken as 1.00.

§ Relative retention time for the corresponding compound on SE-30 taken as 1.00.

ters^{10–12}, i.e., the isomers are eluted from a low-polarity SE-30 column in order of their degree of chlorination (Fig. 5) and that on a polar column the elution order between the 2,2-di- and 2,2,2-trichloroethyl esters is changed (Fig. 6). The effect of chlorine substitution in the alkyl chain seems to be higher with the 4-nitro esters than

TABLE III
RETENTION DATA FOR ETHYL AND ω -CHLOROETHYL ESTERS OF BENZOIC, 4-NITROBENZOIC AND 3,5-DINITROBENZOIC ACIDS, DETERMINED ON SE-30 AND OV-351 CAPILLARY COLUMNS WITH TEMPERATURE PROGRAMMING
Conditions as in Figs. 5 and 6.

Peak No.	Ester	Column		OV-351								
		SE-30		ART*	RRT**	RRT***	RRT§	RRT**	RRT***	RRT§	RRT§§	
<i>Benzoate</i>												
15	Ethyl	4.21		0.52	1.00	1.00	1.00	4.06	2.28	1.00	1.00	0.96
16	2-Chloroethyl	7.55		0.93	1.00	1.00	1.79	10.66	5.99	1.00	2.63	1.41
17	2,2-Dichloroethyl	9.13		1.13	1.00	1.00	2.17	12.16	6.83	1.00	3.00	1.33
18	2,2,2-Trichloroethyl	10.44		1.29	1.00	1.00	2.48	11.62	6.53	1.00	2.86	1.11
<i>4-Nitrobenzoate</i>												
4/15	Ethyl	9.83		1.21	2.33	1.00	1.00	13.35	7.50	3.29	1.00	1.36
4/16	2-Chloroethyl	14.41		1.78	1.91	1.47	1.47	21.04	11.82	1.97	1.58	1.46
4/17	2,2-Dichloroethyl	16.03		1.98	1.76	1.63	1.63	23.02	12.93	1.89	1.72	1.44
4/18	2,2,2-Trichloroethyl	17.12		2.11	1.64	1.74	1.74	21.75	12.22	1.87	1.63	1.27
<i>3,5-Dinitrobenzoate</i> §§§												
35/15	Ethyl	14.88		1.84	3.53	1.00	1.00	—	—	—	—	—
35/16	2-Chloroethyl	19.19		2.37	2.54	1.29	1.29	—	—	—	—	—
35/17	2,2-Dichloroethyl	20.58		2.54	2.25	1.38	1.38	—	—	—	—	—
35/18	2,2,2-Trichloroethyl	21.30		2.63	2.04	1.43	1.43	—	—	—	—	—
C ₁₄	<i>n</i> -Tetradecane	8.10		1.00	—	—	—	1.78	1.00	—	—	0.22

* Absolute retention times (min) were measured from the time of sample injection (Figs. 5 and 6).

** Relative retention time for *n*-tetradecane (C₁₄) taken as 1.00.

*** Relative retention time for the corresponding ester of benzoic acid (15-18) taken as 1.00.

§ Relative retention time for the corresponding ethyl ester (15, 4/15 and 35/15) taken as 1.00.

§§ Relative retention time for the corresponding compound on SE-30 taken as 1.00.

§§§ Esters not analysed on OV-351.

TABLE IV

SUMMARY OF RELATIVE RETENTION TIMES PRESENTED IN TABLES I-III, TOGETHER WITH THE VALUES OBTAINED PREVIOUSLY FOR 4-CHLOROBENZOATES

Ester	Column		
	SE-30	OV-351	
	RRT*	RRT*	RRT**
<i>4-Chlorobenzoate</i>			
Branched-chain***	1.26-1.39	1.23-1.32	1.06-1.16
Unsaturated§	1.21-1.35	1.18-1.27	1.12-1.55
ω -Chloroethyl§§	1.22-1.28	1.18-1.20	1.30-1.50
<i>4-Nitrobenzoate</i>			
Branched-chain	1.78-2.22	2.29-3.27	1.11-1.24
Unsaturated	1.58-2.08	1.89-2.65	1.12-1.59
ω -Chloroethyl	1.64-1.91	1.87-1.97	1.27-1.46
<i>3,5-Dinitrobenzoate</i>			
Branched-chain	2.29-3.22	3.13-4.99	1.15-1.30
Unsaturated	1.95-2.96	2.67-3.74	1.21-1.65
ω -Chloroethyl	2.04-2.54	—	—

* Relative retention time for the corresponding ester of benzoic acid taken as 1.00.

** Relative retention time for the compounds on SE-30 taken as 1.00.

*** From ref. 5.

§ From ref. 9.

§§ From ref. 10.

that with the 4-chloro esters¹⁰, shown from the higher relative retention times, relative to the corresponding ethyl ester (Table III).

A summary of the retention times relative to the parent esters and the compounds on SE-30 is shown in Table IV. The values obtained previously for the corresponding 4-chloro esters^{5,9,10} are included for comparison. On both columns the retention is markedly increased with the series 4-chloro < 4-nitro < 3,5-dinitro ester (Fig. 7), the saturated branched-chain esters showing always higher values than the unsaturated and ω -chloroethyl esters. The ratio between the retention times on the polar and low-polarity phases increases with increasing unsaturation and chlorine substitution, however, as is evident in Tables I-IV.

The results are in accord with those reported previously for the corresponding aliphatic^{1-4,7,8,11,12} and aromatic esters^{5,6,9,10} and *n*-alkyl 4-nitro- and 3,5-dinitrobenzoates¹³. The effect of nitro substitution on the aromatic acyl chain, however, seems to be much more pronounced than that of halogen substitution, as shown by the mutual separation or the reversed retention order of the isomers, preferentially of the unsaturated 3,5-dinitro isomers on a highly polar OV-351 capillary column.

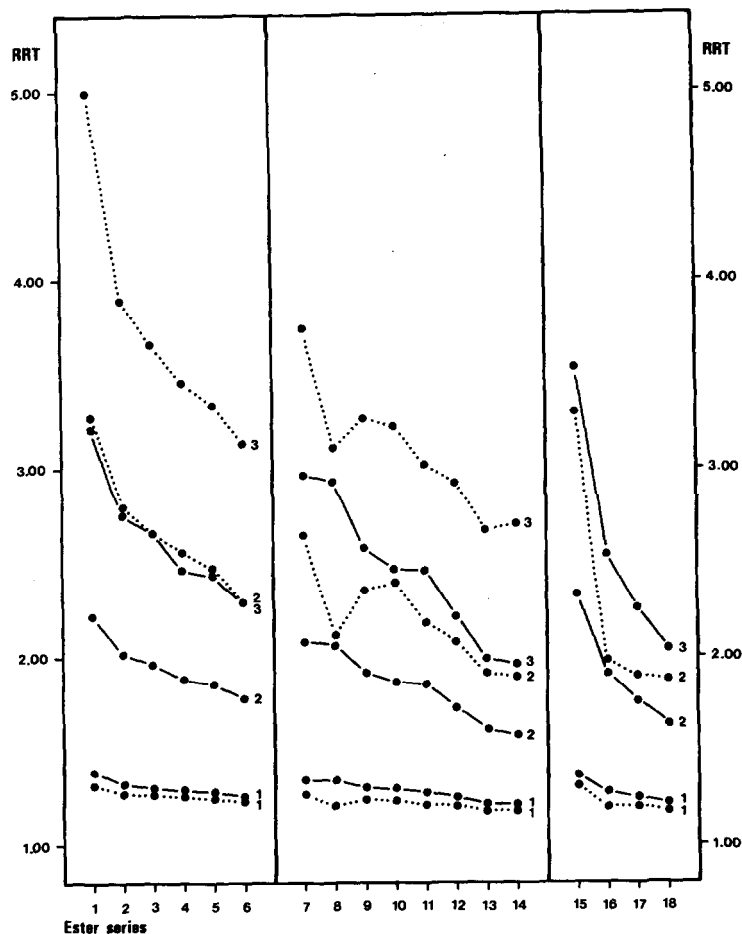


Fig. 7. Plot showing relative retentions of saturated branched-chain (1-6), unsaturated (7-14) and ethyl and ω -chloroethyl (15-18) esters of 4-chlorobenzoic^{5,9,10} (curve 1), 4-nitrobenzoic (curve 2) and 3,5-dinitrobenzoic (curve 3) acids, obtained on SE-30 (●—●) and OV-351 (●····●) capillary columns with operating conditions as shown in Figs. 1-6 and refs. 5, 9 and 10. Relative retention time (RRT) for the corresponding ester of benzoic acid taken as 1.00. Esters 1-18 are listed in Tables I-III.

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