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GAS-LIQUID CHROMATOGRAPHY OF DISUBSTITUTED BENZENE ISOMERS

II. SEPARATION AND STUDY OF THE HALONITROBENZENES, ANISOLES AND TOLUENES

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SUMMARY

Quantitative separation of halonitrobenzenes, anisoles and toluenes was studied on polyethyleneglycol, polyethyleneglycol succinate, polyoxyethylenesorbitan monostearate, silicone oil and squalane as liquid phases. The interactions and forces that affected the separation of the isomers and their elution sequences were discussed. It was found that polyethyleneglycol succinate and polyoxyethylenesorbitan monostearate could selectively separate all the isomers studied.

INTRODUCTION

The separation and study of the dichlorobenzene isomers were presented in Part I (ref. 1). The present work concerns the study of fluoro-, chloro-, and bromo-nitrobenzenes, fluoro-, chloro-, bromo-, and nitroanisoles, and fluoro-, chloro-, bromo-, iodo-, cyano-, and nitrotoluenes. HABBOUSH AND NORMAN² recorded the retention data for nineteen sets of *o*-, *m*-, and *p*-isomers on Apiezon L, dinonyl phthalate, polyethyleneglycol-stearic acid, tritoyl phosphate, 2,4,7-trinitrofluorenone and silicone gum rubber. Nitrotoluenes were resolved by NORMAN³ on 2,4,7-trinitrofluorenone, by COURTIER *et al.*⁴ on cyanated silicone oil, and by PARSON *et al.*⁵ using Apiezon L. BOMBAUGH⁶ examined chloronitrobenzene on Carbowax (1000) and other liquid phases. RATUSKY AND BASTER⁷ studied chloronitrobenzenes, nitroanisoles and other solutes on 1,2,3,4-tetrakis(2-cyanoethoxy)butane.

Most of the reported work did not discuss mechanisms for the separations and interactions involved, but few suggestions for the efficiencies of some systems were given. General quantitative separations and mechanisms of interactions for the isomers dealt with in this study are not available.

The selectivity of stationary liquid phases was studied by several workers⁸⁻¹³, and others¹⁴⁻²⁰ discussed the role of stationary phases in gas-liquid chromatography (GLC), aiming to evaluate quantitatively the interactions of the solutes with liquid

TABLE I
SPECIFIC RETENTION VOLUMES, V_g^0 (ml/g), FOR DISUBSTITUTED BENZENE ISOMERS AT DIFFERENT COLUMN TEMPERATURES

Isomer	PEGS ^a	PA	PEG	SO	SQ
<i>Temperature</i> (°C)	(140)	(120)	(120)	(120)	(140)
<i>o</i> -Fluoronitrobenzene	931	1017	319	538	1174
<i>m</i> -Fluoronitrobenzene	483	509	168	384	617
<i>p</i> -Fluoronitrobenzene	615	644	213	423	760
<i>Temperature</i> (°C)	(160)	(140)	(140)	(120)	(140)
<i>o</i> -Chloronitrobenzene	944	758	283	NR ^b	NE ^c
<i>m</i> -Chloronitrobenzene	673	538	210	NR	NE
<i>p</i> -Chloronitrobenzene	778	612	228	NR	NE
<i>Temperature</i> (°C)	(160)	(140)	(140)	(120)	(140)
<i>o</i> -Bromonitrobenzene	1564	1038	405	NR	NE
<i>m</i> -Bromonitrobenzene	1119	754	302	NR	NE
<i>p</i> -Bromonitrobenzene	1305	850	328	NR	NE
<i>Temperature</i> (°C)	(80)	(100)	(100)	(120)	(140)
<i>o</i> -Fluoroanisole	658	184	58	216	276
<i>m</i> -Fluoroanisole	462	135	46	201	215
<i>p</i> -Fluoroanisole	509	150	49	201	233
<i>Temperature</i> (°C)	(120)	(100)	(100)	(120)	(140)
<i>o</i> -Chloroanisole	516	682	157	632	1001
<i>m</i> -Chloroanisole	348	450	111	561	706
<i>p</i> -Chloroanisole	400	523	128	595	812
<i>Temperature</i> (°C)	(120)	(140)	(100)	(120)	(140)
<i>o</i> -Bromoanisole	905	580	263	1017	881
<i>m</i> -Bromoanisole	629	415	193	928	668
<i>p</i> -Bromoanisole	727	482	226	928	767
<i>Temperature</i> (°C)	(160)	(150)	(140)	(140)	(140)
<i>o</i> -Nitroanisole	2622	1328	456	1218	NE
<i>m</i> -Nitroanisole	1438	703	214	1061	NE
<i>p</i> -Nitroanisole	2859	1473	502	1572	NE

TABLE II

COLUMN EFFICIENCIES FOR DISUBSTITUED BENZENE ISOMERS AT DIFFERENT COLUMN TEMPERATURES

Where a set of isomers was examined at more than one temperature, values of n , α , and η are recorded for the temperature at which resolution was more satisfactory.

Isomer	PEGS			PA			PEG			SO			SQ		
	n	α	η	n	α	η	n	α	η	n	α	η	n	α	η
<i>Temperature</i> (°C)	(140)			(120)			(120)			(120)			(140)		
<i>m</i> -Fluoronitrobenzene	2560	1.27	$1 \cdot 10^{-10}$	2270	1.26	$1 \cdot 10^{-9}$	352	1.27	$2 \cdot 10^{-3}$	6050	1.1	$5 \cdot 10^{-5}$	1550	1.23	10^{-6}
<i>p</i> -Fluoronitrobenzene	2670	1.51	$\ll 10^{-11}$	2800	1.58	$\ll 10^{-11}$	600	1.5	$8 \cdot 10^{-8}$	6550	1.27	$< 10^{-11}$	2050	1.55	$< 10^{-11}$
<i>o</i> -Fluoronitrobenzene	3800			2500			380			5100			1710		
<i>Temperature</i> (°C)	(160)			(140)			(140)								
<i>m</i> -Chloronitrobenzene	1600	1.15	$4 \cdot 10^{-4}$	1950	1.14	$1 \cdot 10^{-3}$	175	1.09	$> 10^{-1}$	NR ^a			NE ^b		
<i>p</i> -Chloronitrobenzene	2210	1.22	$6 \cdot 10^{-6}$	2200	1.24	$7 \cdot 10^{-7}$	270	1.24	$5 \cdot 10^{-2}$	NR			NE		
<i>o</i> -Chloronitrobenzene	2510			2530			600			NR			NE		
<i>Temperature</i> (°C)	(160)			(140)			(140)								
<i>m</i> -Bromonitrobenzene	2650	1.16	$6 \cdot 10^{-5}$	2240	1.13	$5 \cdot 10^{-4}$	435	1.08	$> 10^{-1}$	NR			NE		
<i>p</i> -Bromonitrobenzene	2380	1.2	$8 \cdot 10^{-6}$	3000	1.22	$5 \cdot 10^{-8}$	890	1.24	$5 \cdot 10^{-4}$	NR			NE		
<i>o</i> -Bromonitrobenzene	4000			4100			970			NR			NE		
<i>Temperature</i> (°C)	(80)			(100)			(100)			(120)			(120)		
<i>m</i> -Fluoroanisole	1870	1.13	$1 \cdot 10^{-3}$	1520	1.1	$3 \cdot 10^{-2}$	1025	1.07	$> 10^{-1}$	2150	1.0		1380	1.08	$> 10^{-1}$
<i>p</i> -Fluoroanisole	2140	1.29	10^{-9}	1580	1.23	$5 \cdot 10^{-3}$	700	1.16	$3 \cdot 10^{-2}$	2150	1.07	10^{-2}	960	1.18	$5 \cdot 10^{-3}$
<i>o</i> -Fluoroanisole	3200			2070			1600			3840			1250		
<i>Temperature</i> (°C)	(120)			(100)			(100)			(120)			(120)		
<i>m</i> -Chloroanisole	2720	1.15	$5 \cdot 10^{-5}$	1970	1.16	$2 \cdot 10^{-3}$	455	1.16	10^{-2}	3060	1.06	$2 \cdot 10^{-3}$	1210	1.15	$1 \cdot 10^{-3}$

<i>p</i> -Chloroanisole	3040	1.29	10 ⁻¹²	1600	1.3	5 · 10 ⁻⁸	815	1.22	2 · 10 ⁻³	4670	1.06	2 · 10 ⁻²	1800	1.23	5 · 10 ⁻⁶
<i>o</i> -Chloroanisole	3100			1700			1160			6070			1650		
<i>Temperature</i> (°C)	(120)			(120)			(100)			(120)			(140)		
<i>m</i> -Bromoanisole	2060	1.15	2 · 10 ⁻⁴	2000	1.16	6 · 10 ⁻⁴	364	1.17	3 · 10 ⁻²	1490	1.0		1530	1.15	3 · 10 ⁻³
<i>p</i> -Bromoanisole	2620	1.25	10 ⁻⁸	1820	1.2	5 · 10 ⁻⁵	675	1.16	4 · 10 ⁻²	1490	1.1	3 · 10 ⁻²	1760	1.15	5 · 10 ⁻⁴
<i>o</i> -Bromoanisole	3900			1740			1120			1520			2340		
<i>Temperature</i> (°C)	(160)			(150)			(140)			(140)			NE		
<i>m</i> -Nitroanisole	3470	1.82	≪ 10 ⁻¹⁴	2660	1.89	≪ 10 ⁻¹⁴	424	2.12	10 ⁻¹¹	3170	1.15	5 · 10 ⁻⁷	NE		
<i>o</i> -Nitroanisole	6750	1.09	2 · 10 ⁻⁴	2320	1.11	7 · 10 ⁻³	342	1.1	> 10 ⁻¹	4900	1.29	< 10 ⁻¹⁴	NE		
<i>p</i> -Nitroanisole	9750			3600			468			4080			NE		
<i>Temperature</i> (°C)	(80)			NR			NR			NR			NR		
<i>o</i> -Fluorotoluene	1710	1.09	7 · 10 ⁻²	NR			NR			NR			NR		
<i>m</i> -Fluorotoluene	1025	1.0		NR			NR			NR			NR		
<i>p</i> -Fluorotoluene	1025			NR			NR			NR			NR		
<i>Temperature</i> (°C)	(80)			(80)			(100)			NR			(120)		
<i>o</i> -Chlorotoluene	1370	1.12	8 · 10 ⁻³	2239	1.11	6 · 10 ⁻³	675	1.15	3 · 10 ⁻²	NR			2610	1.09	7 · 10 ⁻²
<i>m</i> -Chlorotoluene	1760	1.0		2250	1.0		710	1.0		NR			1300	1.0	
<i>p</i> -Chlorotoluene	1760			2250			710			NR			1300		
<i>Temperature</i> (°C)	(100)			(120)			(100)			NR			(120)		
<i>o</i> -Bromotoluene	2045	1.1	10 ⁻²	1670	1.1	10 ⁻²	975	1.11	> 10 ⁻¹	NR			3070	1.07	6 · 10 ⁻³

(continued on p. 156)

TABLE II (continued)

Isomer	PEGS			PA			PEG			SO			SQ		
	n	a	η	n	a	η	n	a	η	n	a	η	n	a	η
<i>m</i> -Bromotoluene	2070	1.0		2070	1.0		304	1.11	$>10^{-1}$	NR			5250	1.03	$>10^{-1}$
<i>p</i> -Bromotoluene	2070			2070			375			NR			1110		
Temperature (°C)	(120)			(100)			(100)			NR			NR		
<i>o</i> -Iodotoluene	1870		$>10^{-1}$	1250	1.08	$>10^{-1}$	448	1.1	$>10^{-1}$	NR			NR		
<i>m</i> -Iodotoluene	1600	1.05		825	1.0		225	1.11	$>10^{-1}$	NR			NR		
<i>p</i> -Iodotoluene	1600	1.0		825	1.0		520			NR			NR		
Temperature (°C)	(140)			(120)			(120)			(140)			(140)		
<i>o</i> -Nitrotoluene	3450	1.28	$5 \cdot 10^{-14}$	3660	1.33	$<10^{-14}$	260	1.4	10^{-3}	4000	1.24	10^{-11}	1920	1.33	$<10^{-14}$
<i>m</i> -Nitrotoluene	3520	1.18	$5 \cdot 10^{-7}$	3300	1.18	10^{-6}	320	1.16	$8 \cdot 10^{-2}$	4250	1.09	10^{-3}	3200	1.16	10^{-5}
<i>p</i> -Nitrotoluene	6650			4550			640			6300			3270		
Temperature (°C)	(140)			(120)			(100)			(120)			(140)		
<i>o</i> -Tolunitrile	2100	1.23	$5 \cdot 10^{-8}$	1720	1.27	$5 \cdot 10^{-8}$	940	1.32	$5 \cdot 10^{-7}$	6250	1.14	10^{-10}	1200	1.22	10^{-6}
<i>m</i> -Tolunitrile	2700	1.15	$2 \cdot 10^{-4}$	1900	1.16	$4 \cdot 10^{-4}$	1220	1.15	$8 \cdot 10^{-3}$	7420	1.08	10^{-4}	2240	1.13	10^{-3}
<i>p</i> -Tolunitrile	3770			2300			1680			11750			1700		

^a NR = not resolved.

^b NE = not eluted.

TABLE III

SOME PHYSICAL CONSTANTS FOR DISUBSTITUTED BENZENES

<i>Isomer</i>	n_D^T	<i>T</i> (°C)	<i>E.P.</i> ($cm^3 \times 10^{26}$)	<i>B.p.</i> ^c (°C)	<i>D.M.</i> ^d (Debye)
<i>o</i> -Fluoronitrobenzene	1.5278 ^a	31	12.196	214.6	4.86
<i>m</i> -Fluoronitrobenzene	1.5212 ^a	31	12.068	205.0	3.49
<i>p</i> -Fluoronitrobenzene	1.5269 ^a	31	12.179	205.0	2.53
<i>o</i> -Chloronitrobenzene	1.5330 ^a	85	12.296	245.7	5.01
<i>m</i> -Chloronitrobenzene	1.5385 ^a	85	12.402	235.6	3.47
<i>p</i> -Chloronitrobenzene	1.5501 ^a	85	12.622	242.0	2.43
<i>o</i> -Bromonitrobenzene				261.0	4.97
<i>m</i> -Bromonitrobenzene				256.5	3.48
<i>p</i> -Bromonitrobenzene				256.0	2.45
<i>o</i> -Fluoroanisole	1.4940 ^a	30.5	11.534	155.0	2.32
<i>m</i> -Fluoroanisole	1.4859 ^a	30.5	11.373		1.35
<i>p</i> -Fluoroanisole	1.4830 ^a	30.5	11.313	157.0	0.18
<i>o</i> -Chloroanisole	1.5478 ^b	12.0	12.581	195.0	2.43
<i>m</i> -Chloroanisole	1.5378 ^b	12.0	12.390	194.0	1.65
<i>p</i> -Chloroanisole	1.5401 ^b	12.0	12.434	198.0	0.28
<i>o</i> -Bromoanisole	1.5691 ^a	29.0	12.982	210.0	2.41
<i>m</i> -Bromoanisole	1.5591 ^a	29.0	12.794	211.0	1.41
<i>p</i> -Bromoanisole	1.5596 ^a	29.0	12.802	215.0	0.26
<i>o</i> -Nitroanisole	1.5428 ^a	59	12.483	277.0	4.73
<i>m</i> -Nitroanisole	1.5410 ^a	59	12.446	258.0	3.53
<i>p</i> -Nitroanisole	1.5698 ^a	59	12.994	260.0	2.71
<i>o</i> -Fluorotoluene	1.4703 ^a	20	11.062	114.0	1.28
<i>m</i> -Fluorotoluene	1.4691 ^a	20	11.034	116.0	1.66
<i>p</i> -Fluorotoluene	1.4699 ^a	20	11.054	117.0	1.85
<i>o</i> -Chlorotoluene	1.528 ^b	20	12.200	159.4	1.34
<i>m</i> -Chlorotoluene	1.523 ^b	20	12.106	162.4	1.74
<i>p</i> -Chlorotoluene	1.521 ^b	20	12.064	162.5	1.06
<i>o</i> -Bromotoluene				181.8	1.31
<i>m</i> -Bromotoluene				183.7	1.71
<i>p</i> -Bromotoluene				183.6	1.93
<i>o</i> -Iodotoluene				211.0	1.15
<i>m</i> -Iodotoluene				204.0	1.54
<i>p</i> -Iodotoluene				211.5	1.77
<i>o</i> -Nitrotoluene	1.5488 ^b	15	12.600	222.3	3.83
<i>m</i> -Nitrotoluene	1.5491 ^b	15	12.606	231.0	4.18
<i>p</i> -Nitrotoluene	1.5384 ^b	15	12.401	238.0	4.38
<i>o</i> -Tolunitrile				204.0	3.82
<i>m</i> -Tolunitrile				214.0	4.22
<i>p</i> -Tolunitrile				217.0	4.42

^a Measured in this laboratory.^b Ref. 28 and 29.^c Ref. 30 and 31.^d Calculated as given by SMITH²³.

phases. However, separations of complex mixtures of various chemical compositions were given in the literature, but investigations of their mechanisms of separation were rather rare.

EXPERIMENTAL AND RESULTS

The apparatus, columns and experimental conditions were the same as given in Part I (ref. 1).

Materials

Pure *o*-, *m*- and *p*-fluorotoluenes, chloroanisoles and chlorotoluenes, iodotoluenes, fluoroanisoles and bromoanisoles were obtained from T. J. Sas and Son Ltd., Great Britain. Pure bromotoluenes, nitrotoluenes, tolunitriles, nitroanisoles, *p*-fluoronitrobenzene, chloronitrobenzenes and bromonitrobenzenes were from Hopkin and Williams Ltd., Great Britain. Pure *o*- and *m*-fluoronitrobenzenes were from L. Light and Co. Ltd., Great Britain.

Sampling for liquid isomers was the same as in the previous work, but for solid isomers a 10% (w/w) acetone or ether solution of the isomers was used. Sample sizes ranged from 1–3 μ l.

Specific retention volumes, v_g^0 , for all the isomers on the liquid phases studied were calculated as given by LITTLEWOOD *et al.*²⁷ and are listed in Table I. From the results obtained, the number of theoretical plates, n , relative peak separation, α , and the fractional band impurity, η , were calculated as in Part I. The values of n , α , and η are listed in Table II.

Refractive indices for some isomers were not available; therefore, they were determined in this laboratory in order to calculate the electron polarizabilities per unit volume, α_v^e , as in Part I. An Ape Refractometer fitted with a Colara K0930 ultra-thermostat was used for this purpose. Table III gives some important physical constants (refractive indices, electron polarizabilities, boiling points and dipole moments) for the disubstituted benzene isomers to be utilized in the discussion.

DISCUSSION

The elution order of halonitrobenzenes and of anisoles was always *m*-, *p*- and *o*-. For nitroanisoles, it was *m*-, *o*- and *p*-. These orders are not the order of the increase of the calculated dipole moments of these isomers. The calculated electron polarizabilities per unit volume for these isomers show that the polarization of the *p*-isomers is higher than that of the corresponding *m*-isomer. In fact, *p*-isomers would exert higher polarity than the calculated polarity, this being due to the polarizing effect of the solvent (as shown in Table III).

Toluenes, namely fluoro-, chloro-, bromo-, iodo-, nitro- and cyanotoluenes were eluted in the order: *o*-, *m*- and *p*-. They seem to follow the same order of their calculated dipole moments (as seen in Table III and Fig. 1).

In general, the explanation of the orders can be simplified if one considers the large dipole moment of *p*-isomers, other than toluenes, and the steric effects of the *ortho*-derivatives. The consistently stronger retention of the *p*- versus *m*-isomers seems most likely to be due to an intramolecular electronic effect, as by resonance-

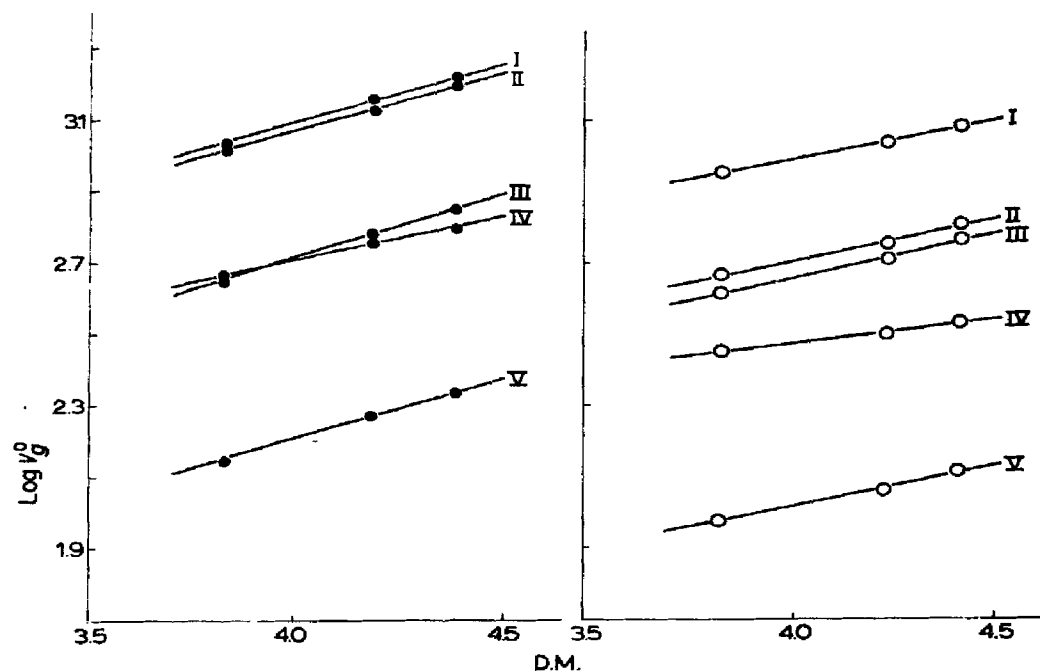


Fig. 1. Variation of $\log V_g^0$ with dipole moment for nitrotoluenes (●) and tolunitriles (○) at 140° and on the following liquid phases: I, Squalane; II, polyethyleneglycol succinate; III, polyoxyethylenesorbitan monostearate; IV, silicone oil; and V, polyethyleneglycol.

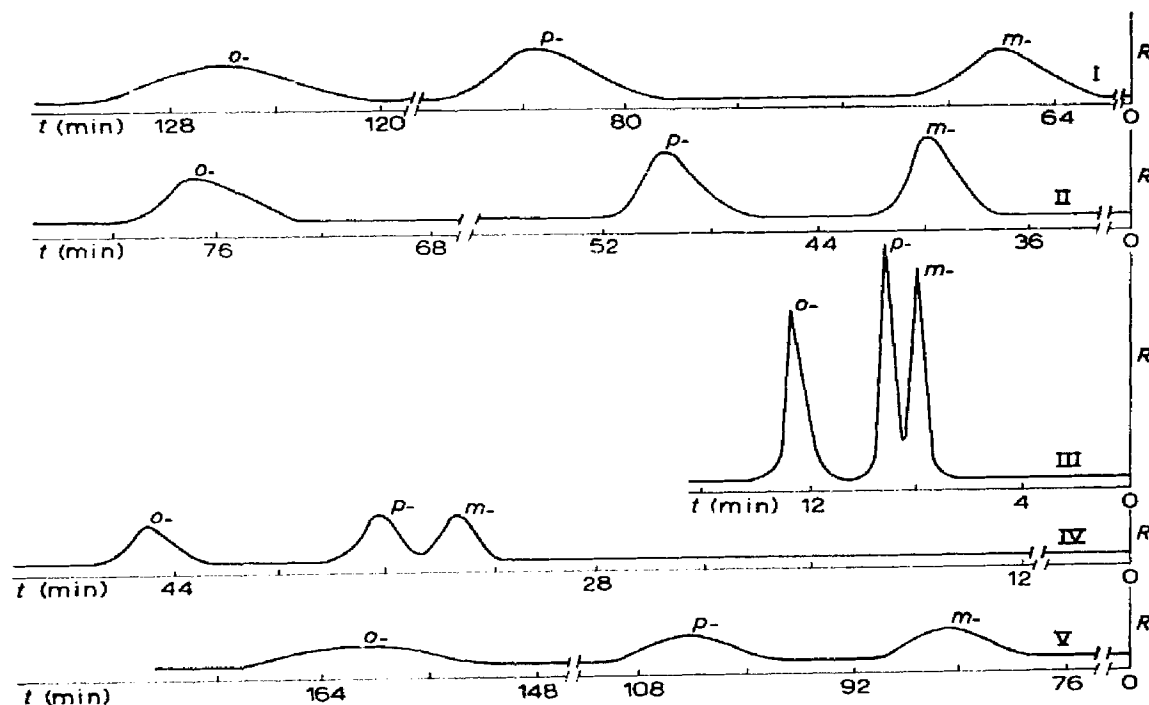


Fig. 2. Chromatograms showing the separation of fluoronitrobenzene isomers on I, polyethyleneglycol succinate at 140° ; II, polyoxyethylenesorbitan monostearate at 140° ; III, polyethyleneglycol at 160° ; IV, silicone oil at 140° ; and V, squalane at 140° . *R* is the recorder deflection and *t* is the retention time in min.

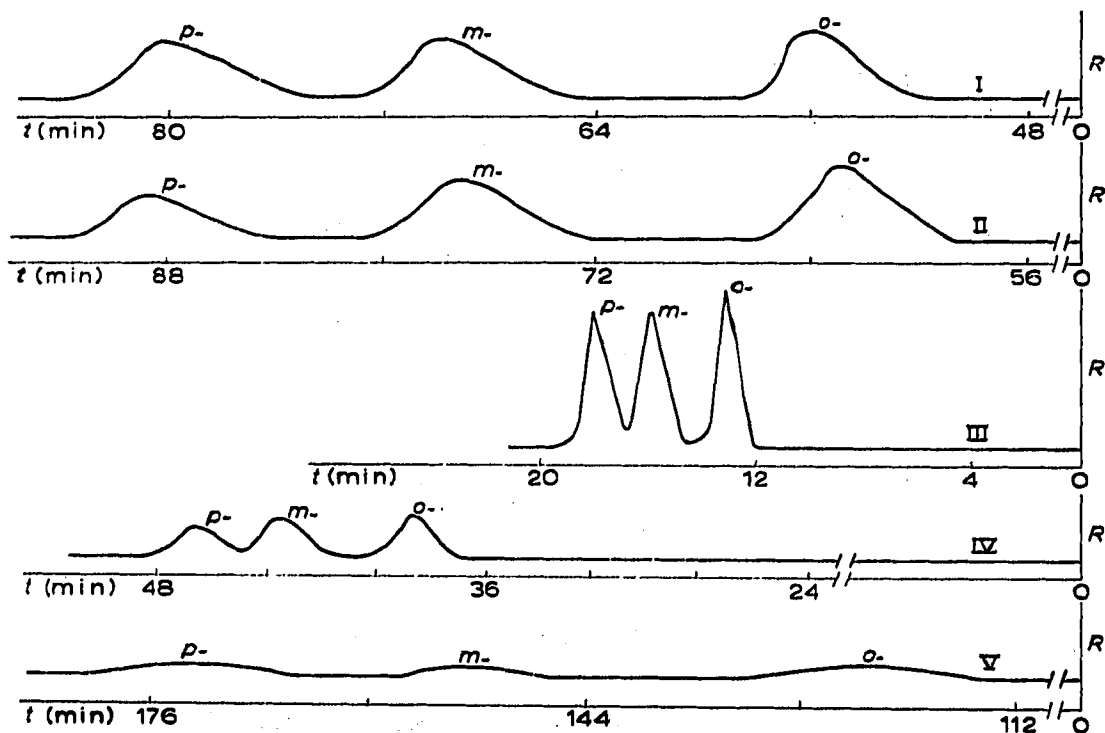


Fig. 3. Chromatograms showing the separation of tolunitriles at 140° on I, polyoxyethylenesorbitan monostearate; II, polyethyleneglycol succinate; III, polyethyleneglycol; IV, silicone oil; V, squalane. R is the recorder deflection and t is the retention time in min.

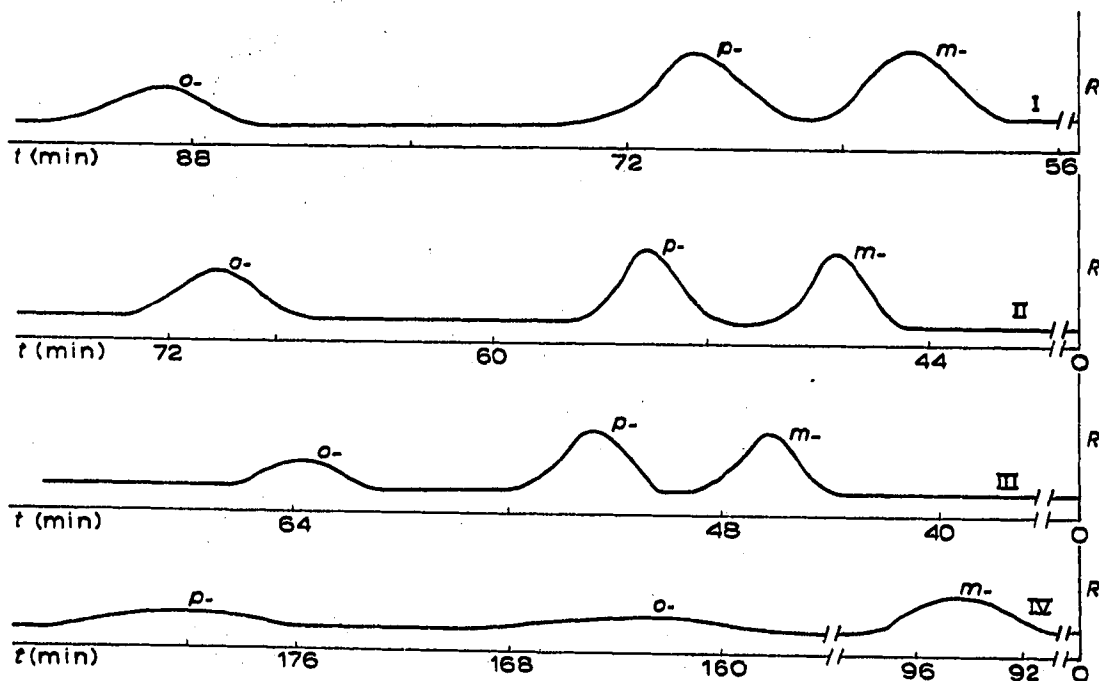


Fig. 4. Chromatograms showing the separation of anisoles on polyethyleneglycol succinate. The chromatograms are represented as follows: I, fluoroanisoles at 80° ; II, chloroanisoles at 120° ; III, bromoanisoles at 140° ; IV, nitroanisoles at 180° . R is the recorder deflection and t is the retention time in min.

induced electron transfer to one of the ring substituents when it is under the influence of a solvent dipole. For toluenes, it seems fairly clear that the steric effects in the *ortho*-derivatives are the main differences. For other derivatives (chloromethoxy, bromo-nitrobenzene, etc.) the *ortho*-derivatives have an effectively larger dipole moment, leading to stronger interactions with the solvent. In the toluenes this effect is missing (alkyl group dipole moments are small) and steric hindrance to solvent interactions with the dipole by the methyl becomes dominant, leading to lesser interaction of the *o*-toluene isomers.

The most selective liquid phases for separation and quantitative determination in this work are the polyesters, *i.e.* PEGS and PA (as seen in Table II, and Figs. 2-4). The substitution of a hydrogen atom in a glycol-type liquid phase by an ester group (succinate or stearate) seems to enhance greatly the selectivity. Carbonyl groups of esters may act as a good polarizing group and would then yield a better resolution of polar solutes.

In SO and SQ, the selectivity was lower than in other liquid phases. They gave wide peaks but were accompanied with larger retention volumes. PEG gave sharp peaks but with a wide base; therefore, the number of theoretical plates was the lowest among the other solvents. In PEG, the specific retention volumes are the smallest among the other liquid phases. This may be caused by the thinner film of the liquid on the solid support due to the higher density of this liquid relative to the other liquids (as in Part I, Table I). In addition, the high molecular weight of PEG and the presence of intermolecular hydrogen bonds would also decrease the retentions of the isomers on this liquid phase.

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