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GAS CHROMATOGRAPHY OF HOMOLOGOUS ESTERS

XXV*. CAPILLARY COLUMN STUDIES OF MONOCHLORINATED C_5–C_{18} *n*-CARBOXYLIC ESTERS

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SUMMARY

The effect on retention of the position of the chlorine substituent in methyl esters of isomeric monochlorinated C_5–C_{18} *n*-carboxylic acids is reported. Incremental effects in terms of retention indices are shown, determined on a non-polar (SE-30) and a polar (Carbowax 20M) capillary column, operated isothermally at temperatures between 100 and 200°C.

INTRODUCTION

The gas chromatographic (GC) separation of the methyl monochloro esters of *n*-carboxylic acids has been the subject of a number of recent studies[1–4], largely directed towards optimizing the resolution of complex reaction mixtures and accordingly using temperature programming on both non-polar (SE-30) and polar (Carbowax 20M or OV-351) capillary columns. The methyl, methyl 2-chloro and chloromethyl esters of C_2–C_{20} *n*-carboxylic acids[1], the *n*-C_1–C_8[2] and *n*-C_9–C_{18}[3] monochloroacetates and the isomeric methyl esters of monochlorinated C_2–C_{18} *n*-carboxylic acids[4] have been separated and the elution orders discussed, while an attempt to determine the effect of the position of the chlorine substituent has been made[5] using data for the methyl 2-chloro ester[1].

The GC retention behaviour, with the retention indices (*I*) and the effect of index increments of the lower (C_2–C_4) chlorinated esters, *i.e.*, *n*-alkyl esters of chlorinated acetic acids[6] and the methyl esters of chlorinated propanoic and butanoic acids[7], on capillary columns has been previously reported.

The present work extends the earlier studies by showing the effect on retention

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of the position of chlorine substitution by a consideration of the incremental effect in terms of retention indices of the isomeric monochloro esters of the C_5–C_{18} *n*-carboxylic acids on a non-polar (SE-30) and a polar (Carbowax 20M) capillary column at temperatures between 100 and 200°C.

EXPERIMENTAL

Materials and methods

The esters were prepared and characterized in the laboratory as previously reported[4].

GC analyses were carried out on a Varian Model 2400 gas chromatograph and on a Perkin-Elmer Sigma 3 instrument under the following operating conditions: a home-made glass capillary column (22 m × 0.30 mm I.D.) coated with 3% Carbowax 20M; a vitreous silica SE-30 wall-coated open-tubular (WCOT) column (25 m × 0.22 mm I.D.), supplied by SGE (North Melbourne, Australia): injector and detector temperatures, 275°C; splitting ratio, 1:20; nitrogen carrier gas flow-rate, 1 ml/min. Retention data were recorded with a Hewlett-Packard Model 3390A reporting integrator.

The retention times were measured from the time of sample injection and the retention indices were determined off-line using a Vector M2 Microprocessor system, the dead volume being first determined by regression analysis from a series of *n*-alkanes using the procedure of Grobler and Balizs[8].

Due to the overlapping, the retention times of the mid-chain isomers of the higher esters are estimates, the isomers being eluted in direct sequence from the 2-chloro to the ω-chloro isomer on both polar and non-polar columns[2–4].

RESULTS AND DISCUSSION

The retention indices of the C_5–C_{18} monochloro esters determined on both SE-30 and Carbowax 20M columns are shown in Table I. Table II shows the retention increments on SE-30 of the chlorine substituent at each position along the acyl chain. The C_5–C_9 esters, with the terminal or near terminal substitution, show at 100°C a considerable enhancement in retention as previously found with chlorine[5] and other substituents[9]. The individual contribution of a particular position of substitution tends to decrease very slightly as the acyl chain length is increased. However, the actual increment at the terminal position is relatively constant. An identical situation occurs at 120 and 140°C where the greatest number of chloro ester series were examined, *i.e.*, the esters from chloropentanoates to chlorotridecanoates, with the individual contributions at any particular position of substitution being slightly increased at the higher temperatures. With further increases in temperature, *i.e.*, from 180 to 200°C, similar behaviour is observed, the retention at the terminal position showing a slight increase with temperature and the incremental increase at any particular position along the chain tending to decrease slightly as the acyl chain length is increased.

The influence on the retention of propanoate and butanoate esters of chlorine and bromine substituents in the acyl chain upon variation of the chain length of the corresponding alcohol has been reported by Komárek *et al.*[10]. The effect of a halogen

TABLE I

RETENTION INDICES OF MONOCHLORO ESTERS OF C_5–C_{18} *n*-ACIDS ON SE-30 AND CARBOWAX 20M

Isomeric methyl ester	*SE-30*					*Carbowax 20M*				
	100°C	*120°C*	*140°C*	*180°C*	*200°C*	*100°C*	*120°C*	*140°C*	*180°C*	*200°C*
Pentanoate	802	654				971	999			
2-Chloro	949	810				1333	1366			
3-Chloro	966	843				1397	1431			
4-Chloro	983	888				1431	1475			
5-Chloro	1045	995				1556	1597			
Hexanoate	904	818				1086	1108			
2-Chloro	1047	988				1436	1470			
3-Chloro	1055	1004				1478	1515			
4-Chloro	1083	1048				1526	1570			
5-Chloro	1095	1065				1571	1615			
6-Chloro	1152	1139				1659	1702			
Heptanoate	1005	962				1241	1233			
2-Chloro	1147	1129				1535	1578			
3-Chloro	1154	1139				1573	1618			
4-Chloro	1176	1166				1604	1650			
5-Chloro	1196	1191				1657	1702			
6-Chloro	1205	1201				1677	1723			
7-Chloro	1257	1260				1756	1799			
Octanoate	1105	1082				1373	1394			
2-Chloro	1248	1251				1635	1676			
3-Chloro	1252	1255				1668	1708			
4-Chloro	1273	1279				1697	1743			
5-Chloro	1284	1291				1731	1774			
6-Chloro	1302	1311				1762	1805			
7-Chloro	1308	1316				1775	1818			
8-Chloro	1358	1369				1851	1893			
Nonanoate	1206	1202	1206			1481	1514	1476		
2-Chloro	1347	1357	1351			1733	1773	1724		
3-Chloro	1351	1362	1354			1765	1805	1755		
4-Chloro	1371	1382	1376			1790	1831	1786		
5-Chloro	1380	1393	1385			1822	1862	1817		
6-Chloro	1391	1403	1396			1837	1877	1834		
7-Chloro	1405	1418	1411			1860	1900	1859		
8-Chloro	1408	1421	1414			1873	1912	1872		
9-Chloro	1459	1472	1465			1947	1985	1946		
Decanoate		1312	1306				1624	1570		
2-Chloro		1459	1451				1869	1820		
3-Chloro		1463	1455				1900	1853		
4-Chloro		1483	1476				1926	1884		
5-Chloro		1491	1484				1954	1912		
6-Chloro		1500	1493				1968	1927		
7-Chloro		1506	1500				1976	1936		
8-Chloro		1520	1514				1998	1959		
9-Chloro		1522	1516				2009	1971		
10-Chloro		1573	1567				2079	2044		

(Continued on p. 92)

TABLE I (*continued*)

Isomeric methyl ester	*SE-30*					*Carbowax 20M*				
	100°C	*120°C*	*140°C*	*180°C*	*200°C*	*100°C*	*120°C*	*140°C*	*180°C*	*200°C*
Undecanoate			1407					1672		
2-Chloro			1551					1917		
3-Chloro			1555					1949		
4-Chloro			1576					1978		
5-Chloro			1583					2006		
6-Chloro			1591					2017		
7-Chloro			1597					2025		
8-Chloro			1602					2033		
9-Chloro			1615					2055		
10-Chloro			1616					2066		
11-Chloro			1668					2139		
Dodecanoate			1508					1770		
2-Chloro			1652					2013		
3-Chloro			1656					2045		
4-Chloro			1677					2074		
5-Chloro			1683					2100		
6-Chloro			1691					2113		
7-Chloro			1696					2117		
8-Chloro			1700					2123		
9-Chloro			1704					2130		
10-Chloro			1718					2152		
11-Chloro			1718					2162		
12-Chloro			1769					2234		
Tridecanoate			1607					1866		
2-Chloro			1752					2110		
3-Chloro			1755					2142		
4-Chloro			1776					2172		
5-Chloro			1783					2197		
6-Chloro			1791					2211		
7-Chloro			1794					2214		
8-Chloro			1796					2218		
9-Chloro			1800					2222		
10-Chloro			1804					2229		
11-Chloro			1817					2249		
12-Chloro			1818					2260		
13-Chloro			1868					2330		
Tetradecanoate				1712					1990	
2-Chloro				1859					2246	
3-Chloro				1863					2279	
4-Chloro				1884					2313	
5-Chloro				1892					2339	
6-Chloro				1899					2353	
7-Chloro				1904					2356	
8-Chloro				1905					2358	
9-Chloro				1907					2359	
10-Chloro				1910					2365	
11-Chloro				1915					2372	
12-Chloro				1929					2396	
13-Chloro				1929					2406	
14-Chloro				1981					2486	

TABLE I (*continued*)

Isomeric methyl ester	*SE-30*					*Carbowax 20M*				
	100°C	*120°C*	*140°C*	*180°C*	*200°C*	*100°C*	*120°C*	*140°C*	*180°C*	*200°C*
Pentadecanoate				1806					2090	
2-Chloro				1957					2349	
3-Chloro				1961					2385	
4-Chloro				1983					2419	
5-Chloro				1990					2448	
6-Chloro				1998					2462	
7-Chloro				2002					2465	
8-Chloro				2002					2468	
9-Chloro				2005					2471	
10-Chloro				2005					2473	
11-Chloro				2009					2478	
12-Chloro				2013					2484	
13-Chloro				2027					2506	
14-Chloro				2028					2517	
15-Chloro				2079					2592	
Hexadecanoate				1902	1902				2191	2202
2-Chloro				2056	2057				2455	2452
3-Chloro				2060	2062				2490	2485
4-Chloro				2083	2083				2522	2520
5-Chloro				2089	2090				2547	2544
6-Chloro				2097	2097				2560	2557
7-Chloro				2100	2099				2562	2559
8-Chloro				2101	2101				2565	2561
9-Chloro				2103	2102				2567	2562
10-Chloro				2104	2103				2570	2564
11-Chloro				2105	2105				2572	2566
12-Chloro				2109	2108				2578	2573
13-Chloro				2113	2113				2584	2579
14-Chloro				2127	2127				2608	2603
15-Chloro				2127	2127				2619	2613
16-Chloro				2181	2178				2696	2691
Heptadecanoate				2011	2007				2295	2297
2-Chloro				2163	2157				2557	2548
3-Chloro				2169	2161				2592	2579
4-Chloro				2192	2183				2628	2614
5-Chloro				2198	2190				2651	2639
6-Chloro				2210	2198				2662	2652
7-Chloro				2214	2201				2664	2653
8-Chloro				2215	2202				2667	2655
9-Chloro				2217	2203				2669	2656
10-Chloro				2218	2204				2672	2657
11-Chloro				2220	2205				2675	2661
12-Chloro				2221	2206				2677	2662
13-Chloro				2222	2210				2686	2668
14-Chloro				2226	2214				2692	2675
15-Chloro				2240	2227				2713	2698
16-Chloro				2240	2228				2725	2709
17-Chloro				2289	2277				2800	2787

(Continued on p. 94)

TABLE I (*continued*)

Isomeric methyl ester	*SE-30*					*Carbowax 20M*				
	100°C	*120°C*	*140°C*	*180°C*	*200°C*	*100°C*	*120°C*	*140°C*	*180°C*	*200°C*
Octadecanoate				2114	2102				2396	2389
2-Chloro				2265	2254				2657	2642
3-Chloro				2269	2259				2691	2675
4-Chloro				2291	2282				2724	2711
5-Chloro				2298	2289				2753	2737
6-Chloro				2309	2305				2761	2743
7-Chloro				2312	2308				2763	2746
8-Chloro				2313	2310				2765	2750
9-Chloro				2314	2313				2767	2753
10-Chloro				2315	2314				2769	2756
11-Chloro				2317	2317				2771	2759
12-Chloro				2317	2318				2773	2762
13-Chloro				2319	2320				2775	2765
14-Chloro				2320	2322				2780	2770
15-Chloro				2324	2325				2787	2777
16-Chloro				2338	2337				2809	2799
17-Chloro				2338	2338				2821	2810
18-Chloro				2388	2381				2897	2884

TABLE II

INCREMENTAL EFFECT OF MONOCHLORO SUBSTITUTION AT EACH POSITION ALONG THE CHAIN FOR C_5–C_{18} METHYL ESTERS ON SE-30

Methyl alkanoate	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}	C_{18}
100°C														
2-Chloro	147	143	142	143	141									
3-Chloro	164	151	149	147	145									
4-Chloro	181	179	171	168	165									
5-Chloro	243	191	191	179	174									
6-Chloro		248	200	197	185									
7-Chloro			252	203	199									
8-Chloro				253	202									
9-Chloro					253									
120°C														
2-Chloro	156	170	167	169	155	147								
3-Chloro	189	186	177	173	160	151								
4-Chloro	234	230	204	197	180	171								
5-Chloro	241	247	229	209	191	179								
6-Chloro		321	239	229	201	188								
7-Chloro			298	234	216	194								
8-Chloro				287	219	208								
9-Chloro					270	210								
10-Chloro						261								
11-Chloro														
12-Chloro														
13-Chloro														

TABLE II (*continued*)

Methyl alkanoate	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}	C_{18}
140°C														
2-Chloro					145	145	144	144	145					
3-Chloro					148	149	148	148	148					
4-Chloro					170	170	169	169	169					
5-Chloro					179	178	176	175	176					
6-Chloro					190	187	184	183	184					
7-Chloro					205	194	190	188	187					
8-Chloro					208	208	195	192	189					
9-Chloro					259	210	208	196	193					
10-Chloro						261	209	210	197					
11-Chloro							261	210	210					
12-Chloro								261	211					
13-Chloro									261					
180°C														
2-Chloro										147	151	154	152	151
3-Chloro										151	155	158	158	155
4-Chloro										172	177	181	181	177
5-Chloro										180	184	187	187	184
6-Chloro										187	192	195	199	195
7-Chloro										192	196	198	203	198
8-Chloro										193	196	199	204	199
9-Chloro										195	199	201	206	200
10-Chloro										198	199	202	207	201
11-Chloro										203	203	203	209	203
12-Chloro										217	207	207	210	203
13-Chloro										217	221	211	211	205
14-Chloro										269	222	225	215	206
15-Chloro											273	225	229	210
16-Chloro												279	229	224
17-Chloro													278	224
18-Chloro														274
200°C														
2-Chloro												155	150	152
3-Chloro												160	154	157
4-Chloro												181	176	180
5-Chloro												188	183	187
6-Chloro												195	191	203
7-Chloro												197	194	206
8-Chloro												199	195	208
9-Chloro												200	196	211
10-Chloro												201	197	212
11-Chloro												203	198	215
12-Chloro												206	199	216
13-Chloro												211	203	218
14-Chloro												225	207	220
15-Chloro												225	220	223
16-Chloro												276	221	235
17-Chloro													270	236
18-Chloro														279

atom introduced into alkyl propanoate or *n*-butanoate molecules is most significant for the lower esters. The retention increment for the lower branched- and normal-chain alkyl halogenopropanoates and -butanoates decreases with increasing chain length of the alkyl group. The values of the increments for the higher esters tend to converge towards a constant value, the incremental effect of the 3-halogeno esters being greater than that of the 2-halogeno esters. These findings where comparable, *i.e.*, 2- and 3-substitution, are in agreement with the present work, while the pattern of decreased incremental retention with the acyl chain length is also evident.

Table III shows the corresponding incremental increase in retention with the same isomers on the polar Carbowax 20M column. The same effects as observed with the non-polar column are found, both with regard to the considerable enhancement of retention with substitution in the terminal position and also with the general slight

TABLE III

INCREMENTAL EFFECT OF MONOCHLORO SUBSTITUTION AT EACH POSITION ALONG THE CHAIN FOR C_5–C_{18} METHYL ESTERS ON CARBOWAX 20M

Methyl alkanoate	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}	C_{18}
100°C														
2-Chloro	362	350	294	262	252									
3-Chloro	426	392	332	295	284									
4-Chloro	460	440	363	324	309									
5-Chloro	585	485	416	358	341									
6-Chloro		573	436	389	356									
7-Chloro			515	402	379									
8-Chloro				478	392									
9-Chloro					466									
120°C														
2-Chloro	367	362	345	282	259	245								
3-Chloro	432	407	385	314	291	276								
4-Chloro	476	462	417	349	317	302								
5-Chloro	598	507	469	380	348	330								
6-Chloro		594	490	411	363	344								
7-Chloro			566	424	386	352								
8-Chloro				499	398	374								
9-Chloro					471	385								
10-Chloro						455								
140°C														
2-Chloro					248	250	245	243	244					
3-Chloro					279	283	277	275	276					
4-Chloro					310	314	306	304	306					
5-Chloro					341	342	334	330	331					
6-Chloro					358	357	345	343	345					
7-Chloro					383	366	353	347	348					
8-Chloro					396	389	361	353	352					
9-Chloro					470	401	383	360	356					
10-Chloro						474	394	382	363					
11-Chloro							467	392	383					
12-Chloro								464	394					
13-Chloro									464					

TABLE III (*continued*)

Methyl alkanoate	C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}	C_{18}
180°C														
2-Chloro										256	259	264	262	261
3-Chloro										289	295	299	297	295
4-Chloro										323	329	331	333	328
5-Chloro										349	358	356	356	357
6-Chloro										363	372	369	367	365
7-Chloro										366	375	371	369	367
8-Chloro										368	378	374	372	369
9-Chloro										369	381	376	374	371
10-Chloro										375	383	379	377	373
11-Chloro										382	388	381	380	375
12-Chloro										406	394	387	382	377
13-Chloro										416	416	393	391	379
14-Chloro										496	427	417	397	384
15-Chloro											502	428	418	391
16-Chloro												505	430	413
17-Chloro													505	425
18-Chloro														501
200°C														
2-Chloro												250	251	253
3-Chloro												283	282	286
4-Chloro												318	317	322
5-Chloro												342	342	348
6-Chloro												355	355	354
7-Chloro												357	356	357
8-Chloro												359	358	361
9-Chloro												360	359	364
10-Chloro												362	360	367
11-Chloro												364	364	370
12-Chloro												371	365	373
13-Chloro												377	371	376
14-Chloro												401	378	381
15-Chloro												411	401	388
16-Chloro												489	412	410
17-Chloro													490	421
18-Chloro														495

reduction in the effect on retention of a particular position of substitution as the acyl chain length was increased. It is apparent that the increased retention on the polar phase is approximately double that on the non-polar phase and a substantial increase is to be expected due to the effect of polar forces introduced by the Carbowax phase.

Plots of retention data on SE-30 of a series available at three temperatures, *i.e.*, C_9 esters, at 100, 120 and 140°C, show that minor increases are evident between the 2- and 3-chloro esters. With the other isomers a steady increase in retention occurs up to the $\omega-1$ position, where the rate of increase falls markedly, being greatly enhanced with the ω-substitution. Retention data for the pentadecanoate esters are available at 180°C and again it is apparent that the overall incremental

TABLE IV

SUMMARY OF INCREMENTAL EFFECTS ON SE-30 AND CARBOWAX 20M

Temp. (°C)	*	*SE-30*													
		C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{13}	C_{15}	C_{16}	C_{17}	C_{18}
100	2	147	143	142	143	141									
	ω	243	248	252	253	253									
	$\omega-2$	96	105	110	110	112									
120	2	156	170	167	169	155	147								
	ω	341	321	298	287	270	261								
	$\omega-2$	185	151	131	118	115	114								
140	2					145	145	144	144	145					
	ω					259	261	261	261	261					
	$\omega-2$					114	116	117	117	116					
180	2										147	151		152	151
	ω										269	273	279	278	274
	$\omega-2$										122	122	125	126	123
200	2											154	155	150	152
	ω												276	270	279
	$\omega-2$												121	120	127

* Position of chlorine substitution and the incremental difference ($\omega-2$) between the ω- and 2-chloro isomers.

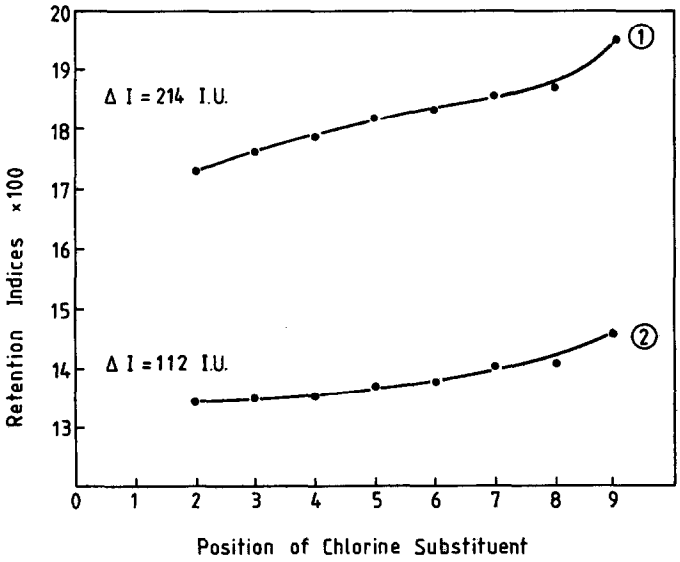

Fig. 1. Plot showing effect of the position of monochloro substitution on retention of nonanoate esters on SE-30 (2) and Carbowax 20M (1) at 100°C. I.U. = Index units.

Carbowax 20M														*Carbowax 20M/SE-30*					
C_5	C_6	C_7	C_8	C_9	C_{10}	C_{11}	C_{12}	C_{13}	C_{14}	C_{15}	C_{16}	C_{17}	C_{18}						
362	350	294	262	252										2.46	2.45	2.07	1.83	1.79	
585	573	515	478	466										2.40	2.31	2.04	1.89	1.84	
223	223	221	216	214										2.32	2.12	2.01	1.96	1.91	
367	362	345	282	259	245									2.35	2.13	2.07	1.67	1.67	1.67
598	594	566	499	471	455									1.76	1.85	1.90	1.74	1.74	1.74
	232	221	217	212	210									1.25	1.54	1.69	1.84	1.84	1.84
				248	250	245	243	244						1.71	1.72	1.70	1.69	1.68	
				470	474	467	464	464						1.81	1.82	1.79	1.78	1.78	
				222	224	222	221	220						1.95	1.93	1.90	1.89	1.90	
									256	259	264	262	261		1.74	1.72	1.71	1.72	1.73
									496	502	505	505	501		1.84	1.84	1.81	1.82	1.83
									240	243	241	243	240		1.97	1.99	1.93	1.93	1.95
											250	251	253				1.61	1.67	1.66
											489	490	495				1.77	1.81	1.77
											239	239	242				1.98	1.99	1.91

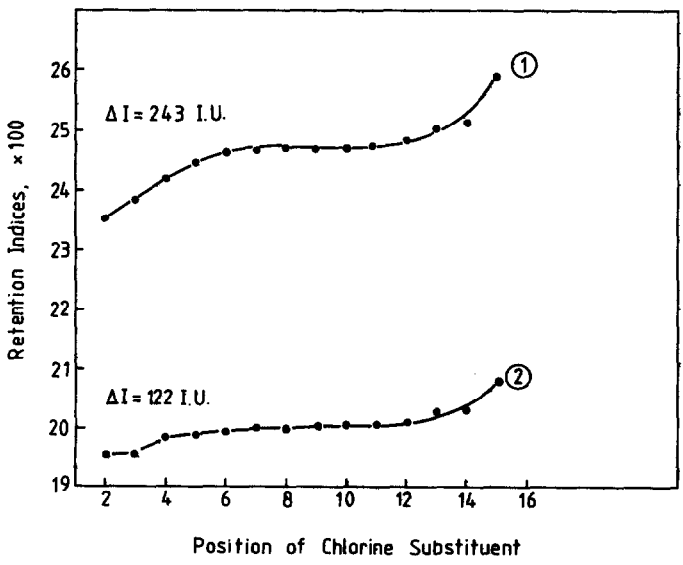

Fig. 2. Plot showing effect of the position of monochloro substitution on retention of pentadecanoate esters on SE-30 (2) and Carbowax 20M (1) at 180°C. I.U. = Index units.

effect is enhanced between the 2- and 3-chloro isomers and reduced between the $\omega-2$ and $\omega-1$ isomers before the ω-position enhancement. On the polar column the effect between the 2- and 3-chloro isomers is not apparent; rather the increase in retention is approximately linear up to the 5-chloro isomer, thereafter it tends to be less apparent, particularly with the longer chain esters until the final considerable enhancement.

The relative effects are shown in Fig. 1 for the nonanoate esters at 100°C, plot 2 showing the effect on SE-30 and plot 1 on Carbowax 20M, and in Fig. 2 for the pentadecanoates at 180°C on the same columns as in plots 2 and 1. With the longer chain esters the same relative effects are apparent, although the mid-chain areas tend to be flatter with the increases more evident near the extremities.

The behaviour of the longer chain esters tends to differ slightly from that of methyl *cis*-undecenoates[11,12] and methyl *cis*- and *trans*-octadecenoates[13,14]. A considerable retention enhancement occurs for these esters at the $\Delta 3$- and $\Delta(\omega-1)$-positions, although the type of variation of the molecule, *i.e.*, double bond of donor character within the chain or a large pendant chlorine atom of acceptor character, is quite different. As with the fatty acid esters[7,15], a reduced incremental effect is evident in the mid-chain areas.

The data from Tables II and III are summarized in Table IV, showing the incremental effect on retention of chlorine at the 2- and ω-positions, together with the incremental differences. At any particular position of substitution all three values are almost doubled on the polar phase, with the absolute increases being reduced at the higher temperatures as might be expected. The values tend to be slightly reduced or become constant at any temperature as the chain length is increased. Overall the values tend to increase with temperature in a regular manner.

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