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GLASS CAPILLARY GAS CHROMATOGRAPHY OF HOMOLOGOUS SERIES OF ESTERS

VI. SEPARATION OF HOMOLOGOUS SERIES OF HALOPROPIONATES OF C_1–C_{10} ALIPHATIC ALCOHOLS AND HALOPROPYL ESTERS OF C_2–C_{10} ALIPHATIC ACIDS OF THE SAME MOLECULAR WEIGHT ON AN SP-400 GLASS CAPILLARY COLUMN

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SUMMARY

The possibility of the gas chromatographic separation and identification of lower and higher halogenated esters of the same molecular weight and empirical formula but differing in their molecular "mirror image", *i.e.*, having the same carbon number and positions of the halogen atoms in the acid chains of halopropionates as in the alcohol chains of halopropyl esters, was studied.

INTRODUCTION

In our previous paper[1] we described the gas chromatographic (GC) separation and identification of halogenated esters and a comparison of the retentions of pairs of halogenated esters with the same molecular weight but differing in their molecular "mirror image", *i.e.*, having the same carbon number and positions of the halogen atoms in the acid chains of haloacetates as in the alcohol chains of 2-haloethyl esters. Because of the imperfect separation of some halogenated esters, capillary GC with temperature programming coupled with mass spectrometry was used[2].

Previously we studied the GC separation of homologous series of halopropionates of C_1–C_{10} aliphatic alcohols and halopropyl esters of C_2–C_{10} aliphatic acids on packed columns under isothermal conditions[3–5].

The systematic GC separation of various types of non-halogenated homologous series of lower esters on packed columns was studied in great detail by Haken and co-workers[6]. More recently, a systematic GC separation of homologous series of various alkyl chloropropionates and chloropropyl esters on glass and silica capillary columns either with temperature programming or under isothermal conditions

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was reported by Korhonen[7–10] and Haken and co-workers[6,11–13], who compared the retentions of homologous esters with the same carbon number in the alcohol as in the acid chains.

In this work, model mixtures of homologous series of C_1–C_{10} *n*-alkyl and C_3–C_5 isoalkyl propionates (Pr), 2-chloropropionates (2-ClPr), 2,2-dichloropropionates (2,2-DClPr), 3-chloropropionates (3-ClPr), *n*-butyrates (Bu) and *n*-propyl esters (PrE), 3-chloropropyl esters (3-ClPrE), 2,3-dichloropropyl esters (2,3-DClPrE), 1,3-dichloroisopropyl esters (1,3-DClisoPrE), isopropyl esters (isoPrE) and *n*-butyl esters (ButE) of *n*-C_2–C_{10} and *iso*-C_4–C_6 aliphatic monocarboxylic acids were separated. Kováts retention indices were used to interpret the results.

EXPERIMENTAL

GC separations were carried out on a Fractovap Model 2150 gas chromatograph (Carlo Erba, Milan, Italy) equipped with a flame ionization detector and an SP-400 glass capillary column. This column was made in our laboratory from soft Unigost soda-lime glass etched with methyl 2-chloro-1,1,2-trifluoroethyl ether vapour, statically silylated with dimethyldichlorosilane vapour and dynamically coated with SP-400 silicone stationary phase. The column efficiency was found to be as follows: capacity ratio, $k = 2.94$; number of theoretical plates per metre, $N = 4020$ for *n*-decane; "Trennzahl", $TZ = 40.5$ for *n*-nonane and *n*-decane.

First, all of the homologous series of lower halogenated esters were separated on a 37 m × 0.16 mm I.D. column, then the column was divided into two lengths of 16 and 21 m. All of the homologous series of the higher halogenated esters were separated on the shorter 16-m column. Nitrogen was used as the carrier gas at a flow-rate of 10–100 cm/s with a splitting ratio of 1:100. The column temperature was maintained at 80°C for the separation of the lower C_1–C_6 *n*-alkyl and C_3–C_5 isoalkyl chloropropionates and chloropropyl esters of *n*-C_2–C_6 monocarboxylic acids, and at 200°C for those of the higher C_6–C_{10} *n*-alkyl chloropropionates and chloropropyl esters of *n*-C_6–C_{10} monocarboxylic acids. The temperatures of the injector and the detector were 250 and 300°C, respectively, for the separation of the higher halogenated esters. The dead time was determined by the injection of methane.

Model mixtures of the lower and higher halogenated esters were obtained from the individual esters. Non-halogenated esters were prepared by the usual sulphuric acid-catalysed esterification and halogenated esters by azeotropic esterification.

RESULTS AND DISCUSSION

The influence of chlorine atoms situated in the alcohol chain of chloropropyl esters of *n*-C_2–C_{10} and *iso*-C_4–C_6 carboxylic acids, the increasing length or branching of the acid carbon chain of esters, the influence of these in the acid chain of C_1–C_{10} *n*-alkyl and C_3–C_5 isoalkyl chloropropionates and the increasing length or branching of the alcohol carbon chain of esters was studied using retention index increments (see Tables I–IV).

Retention index increments, $\Delta I_{\overline{3\text{-Cl}}}$, $\Delta I_{2,3\text{-Cl},\overline{\text{Cl}}}$, $\Delta I_{1,3\text{-Cl},\overline{\text{Cl}}}$*, for chlorine atoms

* The increments with a bar belong to chlorine atoms and methylene groups of the alcohol chain of the esters.

TABLE I

RETENTION INDICES, I, OF C_1–C_6 ALKYL ESTERS AND INCREMENTS OF RETENTION INDICES FOR METHYLENE AND HALOGEN GROUPS AT 80°C

Ester	I	ΔI_{CH_2}	$\Delta I_{2\text{-}Cl}$	$\Delta I_{2,2\text{-}ClCl}$	$\Delta I_{II\text{-}Cl}$	$\Delta I_{3\text{-}Cl}$
PrC_1	626.78	–				
PrC_2	704.69	77.91				
PrC_3	803.78	99.09				
PrC_4	903.37	99.59				
PrC_5	1003.05	99.68				
$PrisoC_3$	748.11	–				
$PrisoC_4$	863.89	115.78				
$PrisoC_5$	966.13	102.24				
2-$ClPrC_1$	782.91	–	156.13			
2-$ClPrC_2$	858.84	75.93	154.15			
2-$ClPrC_3$	952.56	93.72	148.78			
2-$ClPrC_4$	1046.11	93.55	142.74			
2-$ClPrC_5$	1143.56	97.45	140.51			
2-$ClPrisoC_3$	896.75	–	148.64			
2-$ClPrisoC_4$	1009.68	112.93	145.79			
2-$ClPrisoC_5$	1110.34	100.66	144.21			
3-$ClPrC_1$	842.95	–	–	–	–	216.17
3-$ClPrC_2$	920.04	77.09				215.35
3-$ClPrC_3$	1018.24	98.20				215.46
3-$ClPrC_4$	1117.08	98.84				213.71
3-$ClPrC_5$	1215.75	98.67				212.70
3-$ClPrisoC_3$	961.77					213.66
3-$ClPrisoC_4$	1077.45	115.68				213.56
3-$ClPrisoC_5$	1178.40	100.95				212.27
2,2-$DClPrC_1$	864.13	–		237.35	81.22	
2,2-$DClPrC_2$	929.53	65.40		224.84	70.69	
2,2-$DClPrC_3$	1014.63	85.10		210.85	62.07	
2,2-$DClPrC_4$	1109.45	92.82		206.08	63.34	
2,2-$DClPrC_5$	1204.33	94.88		201.28	60.77	
$ButC_1$	717.84	–				
$ButC_2$	795.17	77.33				
$ButC_3$	893.23	98.06				
$ButC_4$	992.26	99.03				
$ButC_5$	1090.42	98.16				
$ButisoC_3$	838.43	–				
$ButisoC_4$	953.82	115.39				
$ButisoC_5$	1055.27	101.45				

were calculated as the differences between the retention indices for 3-chloro- or 2,3-dichloropropyl esters or 1,3-dichloroisopropyl esters and those for corresponding non-halogenated *n*-propyl esters or isopropyl esters, *e.g.*, for the 3-chloropropyl ester of propionic acid, $\Delta I_{\overline{3\text{-}Cl}} = I_{3\text{-}ClPrEC_3} - I_{PrEC_3}$. The retention index increments for chlorine atoms, $\Delta I_{2\text{-}Cl}$ and $\Delta I_{2,2\text{-}Cl,Cl}$, were calculated as the differences between the retention indices for the chloropropionates studied and those for the corresponding

TABLE II

RETENTION INDICES, *I*, OF HALOPROPYL ESTERS OF LOWER CARBOXYLIC ACIDS AND INCREMENTS OF RETENTION INDICES FOR METHYLENE AND HALOGEN GROUPS AT 80°C

Ester	*I*	$\Delta I_{\overline{CH_2}}$	$\Delta I_{\overline{3\text{-}Cl}}$	$\Delta I_{\overline{2,3\text{-}Cl,Cl}}$	$\Delta I_{\overline{1,3\text{-}Cl,Cl}}$
$PrEC_2$	708.12	–			
$PrEC_2$	803.78	95.66			
$PrEC_4$	893.21	89.43			
$PrEC_5$	992.95	99.74			
$PrEC_6$	1092.13	99.18			
$PrEisoC_4$	852.09	–			
$PrEisoC_5$	946.59	94.50			
$PrEisoC_6$	1058.13	111.54			
3-$ClPrEC_2$	946.50	–	238.38		
3-$ClPrEC_3$	1039.89	93.39	236.11		
3-$ClPrEC_4$	1127.92	88.03	234.71		
3-$ClPrEC_5$	1225.70	97.78	232.75		
3-$ClPrEisoC_4$	1083.62	–	231.53		
3-$ClPrEisoC_5$	1178.51	94.89	231.92		
3-$ClPrEisoC_6$	1289.78	111.27	231.65		
2,3-$DClPrEC_2$	1068.23	–		360.11	
2,3-$DClPrEC_3$	1159.32	91.09		355.54	
2,3-$DClPrEC_4$	1245.84	96.52		352.63	
2,3-$DClPrEC_5$	1342.85	97.01		349.90	
2,3-$DClPrEC_6$	1439.03	96.18		346.90	
2,3-$DClPrEisoC_4$	1200.98	–		348.84	
2,3-$DClPrEisoC_5$	1296.27	95.29		349.68	
2,3-$DClPrEisoC_6$	1403.94	107.67		345.81	
$isoPrEC_3$	747.78	–			
$isoPrEC_4$	837.19	89.41			
$isoPrEC_5$	935.82	98.63			
$isoPrEC_6$	1034.05	98.23			
$isoPrEisoC_4$	792.52	–			
$isoPrEisoC_5$	851.09	98.57			
$isoPrEisoC_6$	1000.44	109.55			
1,3-$DClisoPrEC_3$	1138.77	–			390.93
1,3-$DClisoPrEC_4$	1225.19	86.42			388.00
1,3-$DClisoPrEC_5$	1321.34	96.15			385.52
1,3-$DClisoPrEC_6$	1417.52	96.18			383.47
1,3-$DClisoPrEisoC_4$	1178.06	–			385.54
1,3-$DClisoPrEisoC_5$	1276.68	98.62			385.59
1,3-$DClisoPrEisoC_6$	1383.13	106.45			382.69
$ButEC_2$	809.04	–			
$ButEC_3$	903.41	94.37			
$ButEC_4$	992.26	88.85			
$ButEC_5$	1088.90	96.64			
$ButEC_6$	1189.00	100.10			

TABLE III

RETENTION INDICES, I, OF C_6–C_{10} ALKYL ESTERS AND INCREMENTS OF RETENTION INDICES FOR METHYLENE AND HALOGEN GROUPS AT 200°C

Ester	I	ΔI_{CH_2}	$\Delta I_{2\text{-Cl}}$	$\Delta I_{2,2\text{-ClCl}}$	$\Delta I_{II\text{-Cl}}$	$\Delta I_{3\text{-Cl}}$
PrC_6	1103.35	–				
PrC_7	1201.56	98.21				
PrC_8	1298.14	96.58				
PrC_9	1399.44	101.30				
PrC_{10}	1497.70	98.26				
PrC_{12}	1699.60	202.00				
PrC_{14}	1898.60	199.00				
PrC_{16}	2098.09	199.49				
2-$ClPrC_6$	1253.04	–	149.69			
2-$ClPrC_7$	1353.22	100.18	151.66			
2-$ClPrC_8$	1452.09	98.87	153.95			
2-$ClPrC_9$	1559.88	98.79	152.44			
2-$ClPrC_{10}$	1649.76	98.88	152.06			
2,2-$DClPrC_6$	1329.03	–	–	225.68	75.99	
2,2-$DClPrC_7$	1426.56	97.53		225.00	73.33	
2,2-$DClPrC_8$	1524.86	98.30		226.72	72.77	
2,2-$DClPrC_9$	1622.99	98.13		223.55	63.11	
2,2-$DClPrC_{10}$	1722.45	99.46		224.75	72.69	
3-$ClPrC_6$	1324.72					221.47
3-$ClPrC_7$	1424.24	99.52				222.68
3-$ClPrC_8$	1523.35	99.11				225.21
3-$ClPrC_9$	1623.60	100.25				224.16
3-$ClPrC_{10}$	1722.99	99.39				225.29

non-halogenated propionates, *e.g.*, for *n*-propyl 3-chloropropionate, $\Delta I_{3\text{-Cl}} = I_{3\text{-ClPrC}_3} - I_{PrC_3}$.

Retention index increments, $\Delta I_{II2,2\text{-ClCl}}$, for the second chlorine atoms introduced into the 2-chloropropionate molecules were calculated as the differences between retention indices for the 2,2-dichloropropionates studied and those for corresponding 2-chloropropionates, *e.g.*, for ethyl 2,2-dichloropropionate, $\Delta I_{II2,2\text{-ClCl}} = I_{2,2\text{-DClPrC}_2} - I_{2\text{-ClPrC}_2}$.

The retention index increments $\Delta I_{\overline{3\text{-Cl}}}$, $\Delta I_{\overline{2,3\text{-ClCl}}}$, $\Delta I_{\overline{1,3\text{-Cl,Cl}}}$, $\Delta I_{2\text{-Cl}}$, $\Delta I_{3\text{-Cl}}$ and $\Delta I_{2,2\text{-ClCl}}$ decrease with increasing length of the alcohol or acid chains for all the homologous series of the lower halogenated esters. The decreases in these increments are greater and sharper for C_1–C_6 *n*-alkylchloropropionates, whereas those for chloropropyl esters of *n*-C_2–C_6 carboxylic acids are smaller and more gradual. A small and gradual decrease in retention index increments for chlorine atoms is observed for both types of homologous series of lower halogenated esters with branched carbon chains.

The retention increments $\Delta I_{\overline{3\text{-Cl}}}$, $\Delta I_{\overline{2,3\text{-Cl,Cl}}}$, $\Delta I_{\overline{1,3\text{-Cl,Cl}}}$, $\Delta I_{2\text{-Cl}}$, $_{\Delta}I_{2,2\text{-ClCl}}$ and $\Delta I_{3\text{-Cl}}$ for both types of homologous series of higher halogenated esters do not change very much with increasing length of the alcohol or acid chains.

Special attention was paid to halogenated esters with the same molecular

TABLE IV

RETENTION INDICES, *I*, OF HALOPROPYL ESTERS OF HIGHER CARBOXYLIC ACIDS AND INCREMENTS OF RETENTION INDICES FOR METHYLENE AND HALOGEN GROUPS AT 200°C

Ester	*I*	$\Delta I_{\overline{CH_2}}$	$\Delta I_{\overline{3\text{-}Cl}}$	$\Delta I_{\overline{2,3\text{-}Cl,Cl}}$	$\Delta I_{\overline{1,3\text{-}Cl,Cl}}$
$PrEC_6$	1092.98	–			
$PrEC_7$	1191.40	98.52			
$PrEC_8$	1287.80	98.40			
$PrEC_9$	1387.36	99.56			
$PrEC_{10}$	1487.46	100.10			
$PrEC_{12}$	1687.03	199.57			
$PrEC_{14}$	1888.09	201.06			
$PrEC_{16}$	2088.18	200.09			
3-$ClPrEC_6$	1337.77	–	244.79		
3-$ClPrEC_7$	1436.66	98.84	245.26		
3-$ClPrEC_8$	1536.39	99.73	248.52		
3-$ClPrEC_9$	1635.29	98.90	247.93		
3-$ClPrEC_{10}$	1737.21	101.92	249.75		
2,3-$DClPrEC_6$	1464.68	–	–	371.70	
2,3-$DClPrEC_7$	1563.46	98.78	–	372.06	
2,3-$DClPrEC_8$	1662.40	98.94	–	374.60	
2,3-$DClPrEC_9$	1762.16	98.76	–	374.80	
2,3-$DClPrEC_{10}$	1861.09	98.93	–	373.63	
$isoPrEC_6$	1037.56	-			
$isoPrEC_7$	1133.33	95.77			
$isoPrEC_8$	1225.62	92.29			
$isoPrEC_9$	1324.16	98.44			
$isoPrEC_{10}$	1424.13	99.97			
$isoPrEC_{12}$	1622.57	198.44			
$isoPrEC_{14}$	1822.74	200.17			
$isoPrEC_{16}$	2023.31	200.57			
1,3-$DClisoPrEC_6$	1437.17	–			399.61
1,3-$DClisoPrEC_7$	1535.82	98.65			402.49
1,3-$DClisoPrEC_8$	1634.45	99.63			408.73
1,3-$DClisoPrEC_9$	1733.94	99.49			409.78
1,3-$DClisoPrEC_{10}$	1833.40	99.46			409.26
$ButEC_6$	1187.24	–			
$ButEC_7$	1286.02	98.78			
$ButEC_8$	1385.74	99.72			
$ButEC_9$	1484.62	98.88			
$ButEC_{10}$	1584.27	99.65			
$ButEC_{12}$	1783.57	199.30			
$ButEC_{14}$	1984.94	201.37			
$ButEC_{16}$	2185.96	201.02			

weight and empirical formula but differing in their molecular "mirror image" in the carbon number and the positions of chlorine atoms in the alcohol and acid chains. To determine the influence of the number and position of individual chlorine atoms in these types of halogenated esters, we calculated the differences, *D*, between retention indices for pairs of corresponding alkyl chloropropionates and chloropropyl

TABLE V

CALCULATED DIFFERENCES, *D*, IN RETENTION INDICES OF "MIRROR" PAIRS OF LOWER HALOGENATED ESTERS

"Mirror" pair	*Capillary columns*		*Packed columns*			
	OV-101[14,15]	*SP-400*	*Silicone grease*[3–5]	*QF-1*[3–5]	*SE-30*[6]	*Silar 10C*[6]
PrC_2–$PrEC_2$	− 1.8	6.57	− 3	−28	−4.99	−16.11
PrC_3–$PrEC_3$	0.0	0.00	1	3	0	0
PrC_4–$PrEC_4$	10.2	10.16	8	12	−3.21	17.73
PrC_5–$PrEC_5$	10.2	10.10	8	16	−3.15	17.78
$PrisoC_4$–$PrEisoC_4$	10.8	11.80	11	42	–	–
$PrisoC_5$–$PrEisoC_5$	20.0	19.54	19	40	6.64	− 5.4
3-$ClPrC_2$–3-$ClPrEC_2$	−21.9	−26.46	−25	−79		
3-$ClPrC_3$–3-$ClPrEC_3$	−18.5	−21.65	−21	−48		
3-$ClPrC_4$–3-$ClPrEC_4$	−10.3	−10.84	−10	−34		
3-$ClPrC_5$–3-$ClPrEC_5$	− 8.6	− 9.95	−10	−33		
3-$ClPrisoC_4$–3-$ClPrEisoC_4$	− 5.6	− 6.17	− 7	−14		
3-$ClPrisoC_5$–3-$ClPrEisoC_5$	0.2	− 0.11	1	−15		
$ButC_2$–$ButEC_2$	−14.2	−13.87	−12	−60	−7.44	1.11
$ButC_3$–$ButEC_3$	−10.2	−10.18	− 8	−24	−3.21	−17.73
$ButC_4$–$ButEC_4$	0	0	1	− 5	0	0
$ButC_5$–$ButEC_5$	0.4	1.52	0	− 1	−6.2	22.95

TABLE VI

CALCULATED DIFFERENCES, *D*, IN RETENTION INDICES OF "MIRROR" PAIRS OF HIGHER HALOGENATED ESTERS

"Mirror" pair	*Capillary columns*		*Packed columns*	
	OV-101[14,15]	*SP-400*	*Silicone grease*[3–5]	*QF-1*[3–5]
PrC_6–$PrEC_6$	11.2	10.37	4	13
PrC_7–$PrEC_7$	10.2	10.16	9	14
PrC_8–$PrEC_8$	10.1	10.34	8	14
PrC_9–$PrEC_9$	10.0	12.08	11	16
PrC_{10}–$PrEC_{10}$	10.0	10.24	11	11
PrC_{12}–$PrEC_{12}$	–	12.57	10	12
PrC_{14}–$PrEC_{14}$	–	10.51	9	15
PrC_{16}–$PrEC_{16}$	–	9.91	7	10
3-$ClPrC_6$–3-$ClPrEC_6$	− 9.0	−13.05	−16	−38
3-$ClPrC_7$–3-$ClPrEC_7$	− 8.0	−12.42	−13	−42
3-$ClPrC_8$–3-$ClPrEC_8$	− 8.4	−13.04	−12	−44
3-$ClPrC_9$–3-$ClPrEC_9$	− 8.9	−11.63	−12	−42
3-$ClPrC_{10}$–3-$ClPrEC_{10}$	− 8.8	−14.22	−12	−41
$ButC_6$–$ButEC_6$	1.7	0.66	− 5	8
$ButC_7$–$ButEC_7$	0.8	− 0.25	− 4	8
$ButC_8$–$ButEC_8$	1.3	− 0.49	− 4	7
$ButC_9$–$ButEC_9$	1.9	0.53	0	7
$ButC_{10}$–$ButEC_{10}$	2.0	0.90	0	6
$ButC_{12}$–$ButEC_{12}$	–	1.37	− 1	4
$ButC_{14}$–$ButEC_{14}$	–	0.20	− 4	10
$ButC_{16}$–$ButEC_{16}$	–	− 0.30	1	6

esters of carboxylic acids (see Tables V and VI). In these tables, we compare calculated values of *D* with those calculated from our earlier work on packed columns[3–5], on glass capillary columns with OV-101[14,15] and with those calculated from Haken and Srisukh's paper[6], where the lower esters were separated on packed columns.

Comparisons of the *D* values showed that those for non-halogenated "mirror" pairs AcC_x–$EtEC_x$ were 11–16 units[1], whereas those for non-halogenated "mirror" pairs of esters $ButC_x$–$ButEC_x$ were only about 0.2–1.4 units. The differences in *D* for "mirror" pairs of halogenated esters 3-$ClPrC_x$–3-$ClPrEC_x$ are greater then those for "mirror" pairs of non-halogenated esters PrC_x–$PrEC_x$.

These results are in agreement with those of Haken and co-workers[6,12,13] and Korhonen[7,8,10].

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