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GAS-LIQUID CHROMATOGRAPHIC ANALYSES

XIX*. BRANCHED-CHAIN ALCOHOLS AND THEIR PROPANOYL, MONOCHLOROPROPANOYL, BUTANOYL AND MONOCHLOROBUTANOYL DERIVATIVES

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SUMMARY

Branched-chain C₃-C₅ alcohols and their propanoyl, 2-chloropropanoyl, 3-chloropropanoyl, butanoyl, 2-chlorobutanoyl, 3-chlorobutanoyl and 4-chlorobutanoyl derivatives have been analysed by gas chromatography on SE-30 and OV-351 quartz capillary columns with programmed temperature rise. The alcohols and alkyl esters are eluted in the same order on SE-30, whereas some reverse elution orders on OV-351 are observed. The separation of two mixtures, one containing alcohols and their C₃-derivatives and the other containing alcohols and their C₄-derivatives, was better on SE-30, complete resolution of the mixtures not being achieved. The retention order of the compounds is discussed and the results are compared with those of straight-chain alcohols and their derivatives, analysed previously on the same columns under the same operating conditions.

INTRODUCTION

Several papers have been published on the gas chromatography (GC) of alcohols, some of which deal with the GC of lower branched-chain alcohols. Schlunegger¹ separated the mixture of all sixteen possible C₁-C₅ aliphatic alcohols on packed columns coated with 1-nonylphenoxypropane-2,3-diol (NPGA) on Chromosorb A and Bigli *et al.*² studied the GC behaviour of some C₁-C₅ alcohols on Carbowax 400 on Chromosorb P. The correlation between the physical properties and structures of alcohols and their GC behaviour on packed columns coated with polar and non-polar stationary phases has been studied by Castello and co-workers^{3,4}, the application of linear extrathermodynamic relationships to the oxygenated compounds, *e.g.*, to alcohols, having been reported by Haken *et al.*⁵. Drucker⁶ used a wall-coated open-tubular capillary column with SP 1000 as stationary phase for the GC separation of lower straight- and branched-chain alcohols.

* For Part XVIII, see ref. 14.

TABLE I

RETENTION DATA OF BRANCHED-CHAIN ALCOHOLS AND THEIR PROPANOYL, MONOCHLOROPROANOYL, BUTANOYL AND MONOCHLOROBUTANOYL DERIVATIVES, ANALYSED ON SE-30 AND OV-351 QUARTZ CAPILLARY COLUMNS UNDER PROGRAMMED TEMPERATURE RISE

Conditions as shown in Figs. 2-5.

Peak No.	Compound	Column									
		SE-30					OV-351				
		ART*	RRT**	RRT***	RRT§	ART*	RRT**	RRT***	RRT§	RRT§§	RRT§§
1	2-Propanol	2.67	0.12	1.00	—	3.29	0.30	1.00	—	1.23	
2	2-Methyl-2-propanol	2.83	0.13	1.00	—	3.11	0.29	1.00	—	1.10	
3	2-Butanol	3.26	0.15	1.00	—	4.09	0.37	1.00	—	1.25	
4	2-Methyl-1-propanol	3.49	0.16	1.00	—	4.88	0.45	1.00	—	1.40	
5	2-Methyl-2-butanol	3.61	0.17	1.00	—	3.96	0.36	1.00	—	1.10	
6	3-Methyl-2-butanol	4.06	0.19	1.00	—	4.88	0.45	1.00	—	1.20	
7	2-Pentanol	4.29	0.20	1.00	—	5.30	0.49	1.00	—	1.24	
8	3-Methyl-1-butanol	4.80	0.22	1.00	—	6.79	0.62	1.00	—	1.41	
9	Methylethyl propanoate	5.11	0.24	1.91	1.00	3.59	0.33	1.09	1.00	0.70	
10	Dimethylethyl propanoate	5.88	0.27	2.08	1.00	3.59	0.33	1.15	1.00	0.61	
11	1-Methylpropyl propanoate	7.10	0.33	2.18	1.00	4.45	0.41	1.09	1.00	0.63	
12	2-Methylpropyl propanoate	7.50	0.35	2.15	1.00	4.88	0.45	1.00	1.00	0.65	
13	1,1-Dimethylpropyl propanoate	8.31	0.38	2.30	1.00	4.71	0.43	1.19	1.00	0.57	
14	1,2-Dimethylpropyl propanoate	8.90	0.41	2.19	1.00	5.30	0.49	1.09	1.00	0.60	
15	1-Methylbutyl propanoate	9.37	0.43	2.18	1.00	5.63	0.52	1.06	1.00	0.60	
16	3-Methylbutyl propanoate	10.17	0.47	2.12	1.00	6.59	0.60	0.97	1.00	0.65	
17	Methylethyl 2-chloropropanoate	8.11	0.38	3.04	1.59	7.19	0.66	2.19	2.00	0.89	
18	Dimethylethyl 2-chloropropanoate	9.13	0.42	3.23	1.55	7.09	0.65	2.28	1.97	0.78	
19	1-Methylpropyl 2-chloropropanoate	10.75	0.50	3.30	1.51	9.00	0.82	2.20	2.02	0.84	
20	2-Methylpropyl 2-chloropropanoate	11.19	0.52	3.21	1.49	9.68	0.89	1.99	1.98	0.87	
21	1,1-Dimethylpropyl 2-chloropropanoate	12.12	0.56	3.36	1.46	9.38	0.86	2.37	1.99	0.77	
22	1,2-Dimethylpropyl 2-chloropropanoate	12.76	0.59	3.14	1.43	10.25	0.94	2.10	1.93	0.80	
23	1-Methylbutyl 2-chloropropanoate	13.12	0.61	3.06	1.40	10.70	0.98	2.02	1.90	0.82	
24	3-Methylbutyl 2-chloropropanoate	14.00	0.65	2.92	1.38	12.03	1.10	1.77	1.83	0.86	
25	Methylethyl 3-chloropropanoate	9.92	0.46	3.72	1.94	10.35	0.95	3.15	2.88	1.04	
26	Dimethylethyl 3-chloropropanoate	11.09	0.51	3.92	1.89	10.22	0.94	3.29	2.85	0.92	
27	1-Methylpropyl 3-chloropropanoate	12.69	0.59	3.89	1.79	12.39	1.14	3.03	2.78	0.98	
28	2-Methylpropyl 3-chloropropanoate	13.12	0.61	3.76	1.75	13.15	1.21	2.69	2.69	1.00	
29	1,1-Dimethylpropyl 3-chloropropanoate	14.14	0.65	3.92	1.70	12.80	1.17	3.23	2.72	0.91	
30	1,2-Dimethylpropyl 3-chloropropanoate	14.61	0.68	3.60	1.64	13.65	1.25	2.80	2.58	0.93	

31	1-Methylbutyl 3-chloropropanoate	15.01	0.70	3.50	1.60	14.12	1.29	2.66	2.51	0.94
32	3-Methylbutyl 3-chloropropanoate	15.79	0.73	3.29	1.55	15.33	1.41	2.26	2.33	0.97
33	Methylethyl butanoate	6.93	0.32	2.60	1.00	4.24	0.39	1.29	1.00	0.61
34	Dimethylethyl butanoate	7.85	0.36	2.77	1.00	4.24	0.39	1.36	1.00	0.54
35	1-Methylpropyl butanoate	9.39	0.43	2.88	1.00	5.48	0.50	1.34	1.00	0.58
36	2-Methylpropyl butanoate	9.83	0.46	2.82	1.00	5.92	0.54	1.21	1.00	0.60
37	1,1-Dimethylpropyl butanoate	10.71	0.50	2.97	1.00	5.79	0.53	1.46	1.00	0.54
38	1,2-Dimethylpropyl butanoate	11.36	0.53	2.80	1.00	6.46	0.59	1.32	1.00	0.57
39	1-Methylbutyl butanoate	11.80	0.55	2.75	1.00	6.89	0.63	1.30	1.00	0.58
40	3-Methylbutyl butanoate	12.62	0.58	2.63	1.00	7.92	0.73	1.17	1.00	0.63
41	Methylethyl 2-chlorobutanoate	10.60	0.49	3.97	1.53	8.50	0.78	2.58	2.00	0.80
42	Dimethylethyl 2-chlorobutanoate	11.67	0.54	4.12	1.49	8.41	0.77	2.70	1.98	0.72
43	1-Methylpropyl 2-chlorobutanoate	13.35	0.62	4.10	1.42	10.40	0.95	2.54	1.90	0.78
44	2-Methylpropyl 2-chlorobutanoate	13.78	0.64	3.95	1.40	11.03	1.01	2.26	1.86	0.80
45	1,1-Dimethylpropyl 2-chlorobutanoate	14.69	0.68	4.07	1.37	10.79	0.99	2.72	1.86	0.73
46	1,2-Dimethylpropyl 2-chlorobutanoate	15.23	0.71	3.75	1.34	11.52	1.06	2.36	1.78	0.76
47	1-Methylbutyl 2-chlorobutanoate	15.61	0.72	3.64	1.32	12.10	1.11	2.28	1.76	0.78
48	3-Methylbutyl 2-chlorobutanoate	16.39	0.76	3.41	1.30	13.33	1.22	1.96	1.68	0.81
49	Methylethyl 3-chlorobutanoate	11.04	0.51	4.13	1.59	9.73	0.89	2.96	2.29	0.88
50	Dimethylethyl 3-chlorobutanoate	12.21	0.57	4.31	1.56	9.68	0.89	3.11	2.28	0.79
51	1-Methylpropyl 3-chlorobutanoate	13.78	0.64	4.23	1.47	11.73	1.08	2.87	2.14	0.85
52	2-Methylpropyl 3-chlorobutanoate	14.20	0.66	4.07	1.44	12.42	1.14	2.55	2.10	0.87
53	1,1-Dimethylpropyl 3-chlorobutanoate	15.19	0.70	4.21	1.42	12.16	1.11	3.07	2.10	0.80
54	1,2-Dimethylpropyl 3-chlorobutanoate	15.69	0.73	3.86	1.38	13.01	1.19	2.67	2.01	0.83
55	1-Methylbutyl 3-chlorobutanoate	16.04	0.74	3.74	1.36	13.48	1.24	2.54	1.96	0.84
56	3-Methylbutyl 3-chlorobutanoate	16.79	0.78	3.50	1.33	14.61	1.34	2.15	1.84	0.87
57	Methylethyl 4-chlorobutanoate	12.91	0.60	4.84	1.86	12.27	1.12	3.73	2.89	0.95
58	Dimethylethyl 4-chlorobutanoate	14.00	0.65	4.95	1.78	12.11	1.11	3.89	2.86	0.87
59	1-Methylpropyl 4-chlorobutanoate	15.51	0.72	4.76	1.65	14.19	1.30	3.47	2.59	0.91
60	2-Methylpropyl 4-chlorobutanoate	16.00	0.74	4.58	1.63	14.91	1.37	3.06	2.52	0.93
61	1,1-Dimethylpropyl 4-chlorobutanoate	16.79	0.78	4.65	1.57	14.52	1.33	3.67	2.51	0.86
62	1,2-Dimethylpropyl 4-chlorobutanoate	17.31	0.80	4.26	1.52	15.36	1.41	3.15	2.38	0.89
63	1-Methylbutyl 4-chlorobutanoate	17.70	0.82	4.13	1.50	15.80	1.45	2.98	2.29	0.89
64	3-Methylbutyl 4-chlorobutanoate	18.53	0.86	3.86	1.47	17.11	1.57	2.52	2.16	0.92
65	<i>n</i> -Tetradecane	21.59	1.00	—	—	10.91	1.00	—	—	0.51

* Absolute retention times (min) were measured from sample injection (Figs. 2-5).

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

*** Relative retention time for the corresponding alcohol taken as 1.00.

§ Relative retention time for the corresponding non-chlorinated ester taken as 1.00.

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

The GC retention behaviour of a number of series of homologous esters on non-polar and polar stationary phases has been studied widely by Haken and co-workers⁷⁻¹². However, the GC of esters of branched-chain alcohols has been little investigated, since only the results of methylethyl (isopropyl), 2-methylpropyl (isobutyl) and 3-methylbutyl (isoamyl) alkanoates seem to have been reported.

Previously, the GC separations of aliphatic C₁–C₁₈ straight-chain alcohols and the corresponding *n*-alkyl propanoates, butanoates and their monochlorinated derivatives have been reported by the author^{13,14}. The present paper gives the results of lower branched-chain alcohols and their propanoyl, 2-chloropropanoyl, 3-chloropropanoyl, butanoyl, 2-chlorobutanoyl, 3-chlorobutanoyl and 4-chlorobutanoyl derivatives, analysed on SE-30 and OV-351 capillary columns with programmed temperature rise. The relative retention times of the compounds are given and the retention order of the alcohols and alkyl esters is discussed. The results are compared with the retention behaviour of alcohols reported earlier¹⁻⁶, and with the results of lower straight-chain homologues, given in the earlier parts of this series^{13,14}.

EXPERIMENTAL

Materials and methods

Branched-chain alcohols (1-8) and *n*-tetradecane (65) were commercial products (Fluka, Buchs, Switzerland) and were used without further purification. Alkyl propanoates (9-16), 2-chloropropanoates (17-24), 3-chloropropanoates (25-32), butanoates (33-40), 2-chlorobutanoates (41-48), 3-chlorobutanoates (49-56) and 4-chlorobutanoates (57-64) were prepared from the corresponding alcohols and acid chlorides as described earlier^{13,14}. For the numbering of the compounds see Table I.

The GC analyses were performed on a Perkin-Elmer Sigma 3 gas chromatograph under the operating conditions reported previously¹⁴. The column (SE-30 and OV-351) temperature was programmed from 50°C at 6°C min⁻¹ until the elution of peaks had ceased. The chromatographic data were analysed with a Hewlett-Packard Model 3390A reporting integrator, using standard programs.

RESULTS AND DISCUSSION

Fig. 1 shows the correlation between the boiling points and the retention times of branched-chain alcohols studied and of lower straight-chain alcohols reported earlier¹⁴. As shown, alcohols are eluted on SE-30, with three exceptions, in the order of increasing boiling point, *i.e.*, 2-propanol (1) is eluted earlier than ethanol, 2-methyl-1-propanol (4) earlier than 2-methyl-2-butanol (5) and 1-butanol earlier than 3-methyl-2-butanol (6). Correspondingly, the following compound pairs are eluted in the reversed order on a polar OV-351 capillary column: 2-methyl-2-propanol (2) and 2-propanol (1), 2-methyl-2-butanol (5) and 2-butanol (3), 2-pentanol (7) and 1-butanol. In addition, 2-methyl-1-propanol (4) and 3-methyl-2-butanol (6) completely overlap. As shown, a relatively high retention time on OV-351 for 3-methyl-1-butanol (8), as compared with that of 2-pentanol (7), is observed (Fig. 1).

The retention order of the branched-chain alcohols studied is in accordance with that observed earlier on several polar and non-polar packed and capillary col-

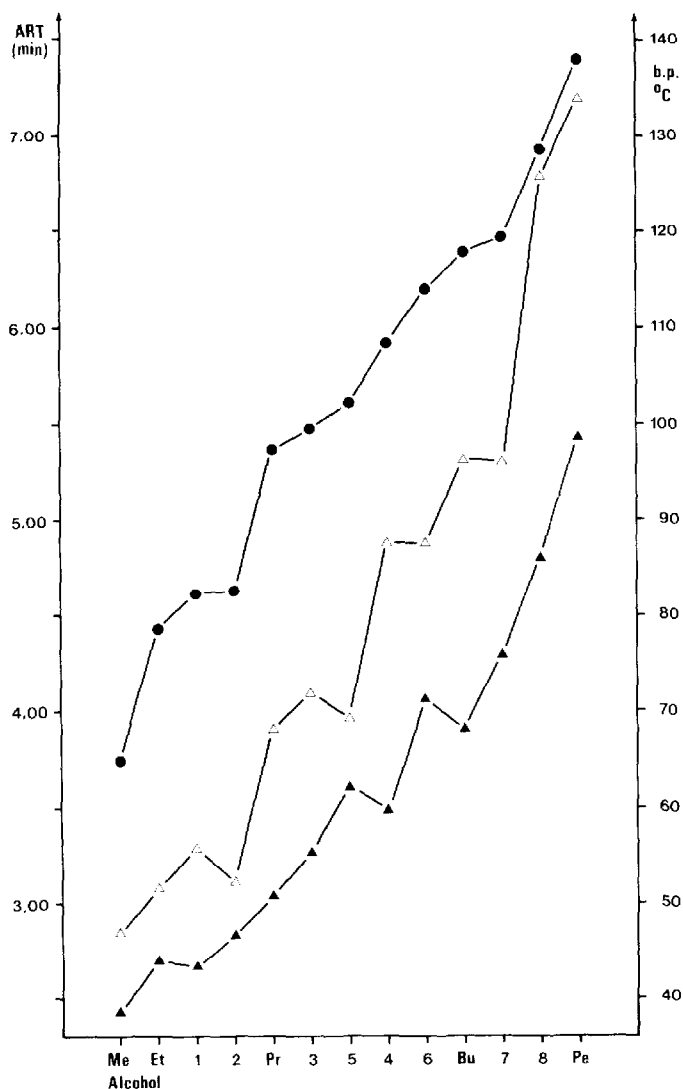


Fig. 1. The correlation between the boiling points (●) and the retention times (ARTs) of the alcohols, analysed on SE-30 (▲) and OV-351 (△). The boiling points (b.p.) are taken from refs. 2 and 3 and the retention times of straight-chain alcohols from ref. 14. Me = Methanol, Et = ethanol, Pr = propanol, Bu = butanol, Pe = pentanol; the alcohols (1-8) are identified in Table I.

umns under isothermal operating conditions²⁻⁶. Only one column (NPGA) has been reported to give a diverging retention order of a mixture of lower alcohols¹. However, straight-chain alcohols are eluted on the non-polar packed columns relatively earlier, giving rise to the reverse elution orders³⁻⁵, whereas on the polar columns this phenomenon is reversed²⁻⁶. It should be also noted that changes in the operating temperature have been shown to effect changes in the retention order of the alcohols studied^{2,6}.

Chromatograms of a mixture of branched-chain alcohols (1-8) and their propanoyl (9-16) and monochloropropanoyl (17-32) derivatives are illustrated in Figs.

2 and 3, which refer to analysis on SE-30 and OV-351, respectively. Figs. 4 and 5 give the corresponding separations of alcohols (1-8) and their butanoyl (33-40) and monochlorobutanoyl (41-64) derivatives. The retention data of the compounds are presented in Table I.

As shown, the esters of branched-chain alcohols are eluted in the same order as the branched-chain alcohols on a non-polar stationary phase (Figs. 2 and 4). As expected, the elution order of the esters is the same as previously reported for the straight-chain homologues¹⁴, *i.e.*, propanoate < butanoate < 2-chloropropanoate < 3-chloropropanoate < 2-chlorobutanoate < 3-chlorobutanoate < 4-chlorobutanoate. All alcohols (1-8) are eluted earlier than methylethyl propanoate (9), with methylethyl 2-chloropropanoate (17) and 3-chloropropanoate (25) being eluted earlier than 1,1-dimethylpropyl propanoate (13) and 3-methylbutyl propanoate (16), respectively. One complete overlapping (compounds 28 and 23) and three partial separated peaks of a mixture of alcohols and their propanoyl derivatives are observed (Fig. 2).

Fig. 4 shows the poorer resolution of a mixture of alkyl butanoates, *i.e.*, compounds 44 and 51, as well as 56 and 61 overlapping. In addition, several partial overlaps are detected. 1,1-Dimethylpropyl butanoate (37) is eluted between methylethyl 2-chlorobutanoate (41) and 3-chlorobutanoate (49), whereas the corresponding ω -chloro isomer (57) appeared later than the last eluted non-chlorinated ester, *i.e.*, 3-methylbutyl butanoate (40). As mentioned above, the corresponding C₃-derivatives (25 and 16) are eluted in the reversed order.

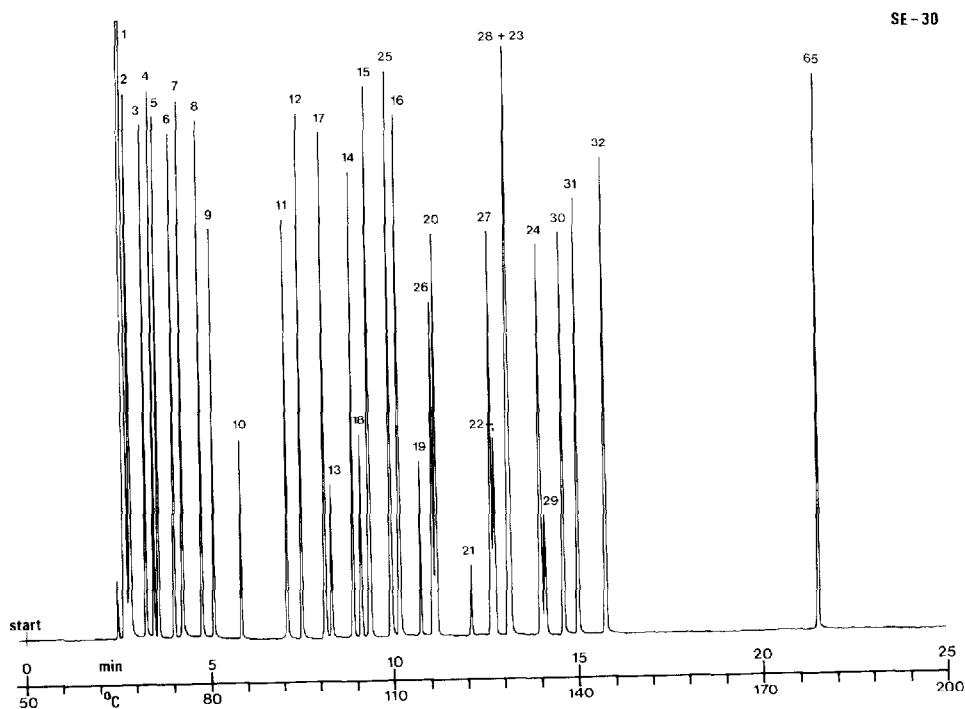


Fig. 2. Chromatogram of a mixture of branched-chain alcohols (1-8) and their propanoyl (9-16) and monochloropropanoyl derivatives (17-32), analysed on SE-30. Compounds are identified in Table I.

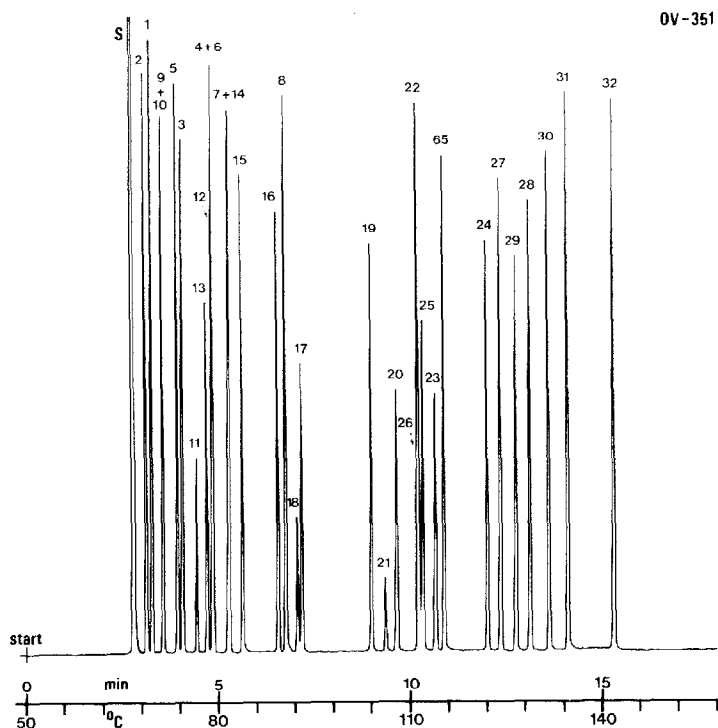


Fig. 3. Chromatogram of a mixture of branched-chain alcohols (1-8) and their propanoyl (9-16) and monochloropropanoyl derivatives (17-32), analysed on OV-351, S = Solvent; compounds are identified in Table I.

By comparing the retention behaviour of the C_3 - and C_4 -derivatives of straight- and branched-chain alcohols, analysed on SE-30, it is evident that the retention order follows that obtained for the alcohols but with one exception, *i.e.*, ethyl esters are eluted earlier than methylethyl esters. However, the difference between the retention times of ethanol¹⁴ and 2-propanol (1) is negligible, the alcohols overlapping on SE-30. In addition, a slightly lower retention time for butyl 3-chloropropanoate¹⁴ relative to 1,1-dimethylpropyl 3-chloropropanoate (29) is observed.

It has been reported previously¹⁵ that the esters of secondary alcohols¹⁶ are eluted in the same order as the secondary alcohols on both polar and non-polar stationary phases, the same elution order occurring also for tertiary alcohols¹⁷. Figs. 3 and 5 show that the esters of 2-methyl-2-butanol (5) and 2-butanol (3) are eluted in the reverse order on OV-351 as compared with the alcohols. In addition, methyl-ethyl and dimethylethyl propanoates (9 and 10) and butanoates (33 and 34) overlap, whereas the corresponding alcohols (2 and 1) are separated and *vice versa*, *i.e.*, esters are separated and alcohols overlap, 2-methyl-1-propanol (4) and 3-methyl-2-butanol (6) are coincident, whereas their derivatives (12,14; 20,22; 28,30; 36,38; 44,46; 52,54; and 60,62) are resolved from each other.

As reported previously¹³, alcohols from methanol to 1-butanol are eluted on OV-351 earlier than their propanoyl derivatives, whereas for longer chain lengths (C_5 - C_{18}) the reverse elution order of the compounds is detected. The branched-chain

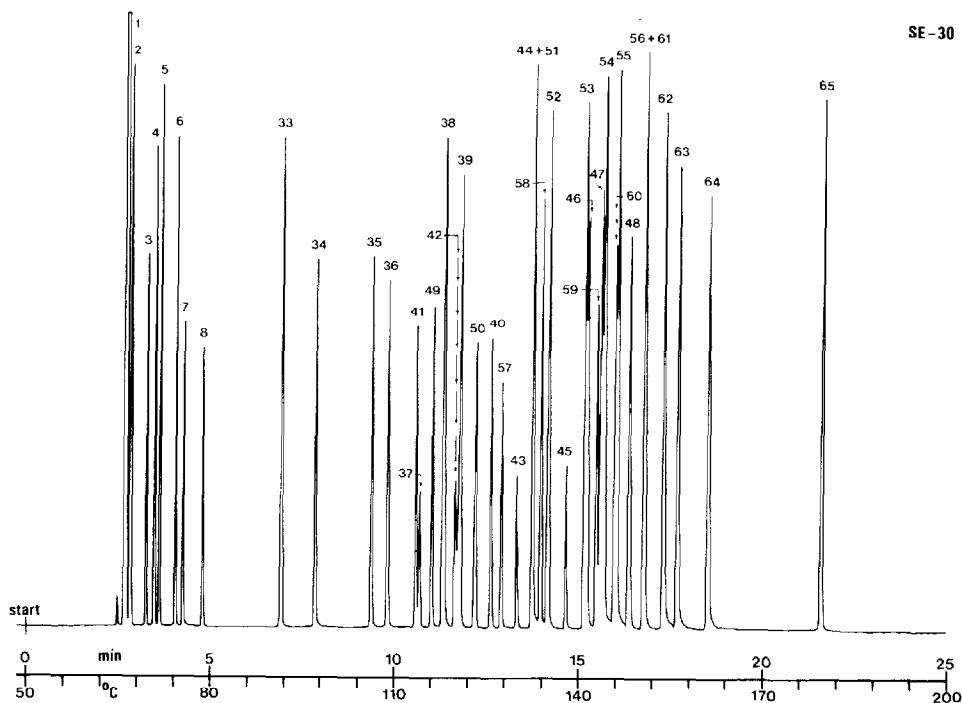


Fig. 4. Chromatogram of a mixture of branched-chain alcohols (1-8) and their butanoyl (33-40) and monochlorobutanoyl derivatives (41-64), analysed on SE-30. Compounds are identified in Table I.

alcohols studied are eluted earlier, except for 2-methyl-1-propanol (4) and 3-methyl-1-butanol (8), than the corresponding alkyl propanoates, which the alcohol (4) and 2-methylpropyl propanoate (12) being coincident and 3-methylbutyl propanoate (16) being eluted earlier than 3-methyl-1-butanol (8), as shown in Fig. 3.

As expected, the homologous esters are eluted on a polar column in the order propanoate < butanoate < 2-chloropropanoate < 2-chlorobutanoate < 3-chlorobutanoate < 3-chloropropanoate < 4-chlorobutanoate¹⁴. Poorer separations of the mixtures are observed on OV-351, the overlaps which occur being shown in Figs. 3 and 5. Two alcohols are eluted earlier than the first eluted propanoyl derivatives (9 and 10) and four before the butanoyl derivatives (33 and 34), all the non-chlorinated esters (9-16 and 33-40) being eluted earlier than the monochloro derivatives (18) and (42). Five 2-chloro esters (17-21) left the column before dimethylethyl ω -chloropropanoate (26), whereas seven 2-chloro isomers (41-47) were eluted earlier than dimethylethyl ω -chlorobutanoate (58).

By comparing the retention behaviour of the monochlorinated *n*-alkyl esters^{13,14} and the branched-chain esters studied in the present paper, the following disparities in retention order compared to that of the corresponding parent esters on OV-351 are observed: ethyl 2- and 3-chloropropanoates are eluted later than the corresponding dimethylethyl (18 and 17) and methylethyl (26 and 25) monochloropropanoates, respectively; ethyl 2-, 3- and 4-chlorobutanoates later than dimethylethyl and methylethyl monochlorobutanoates (42 and 41; 50 and 49; and 58 and 57), respectively; propyl 2-chlorobutanoate later than 1-methylpropyl 2-chlorobutanoate

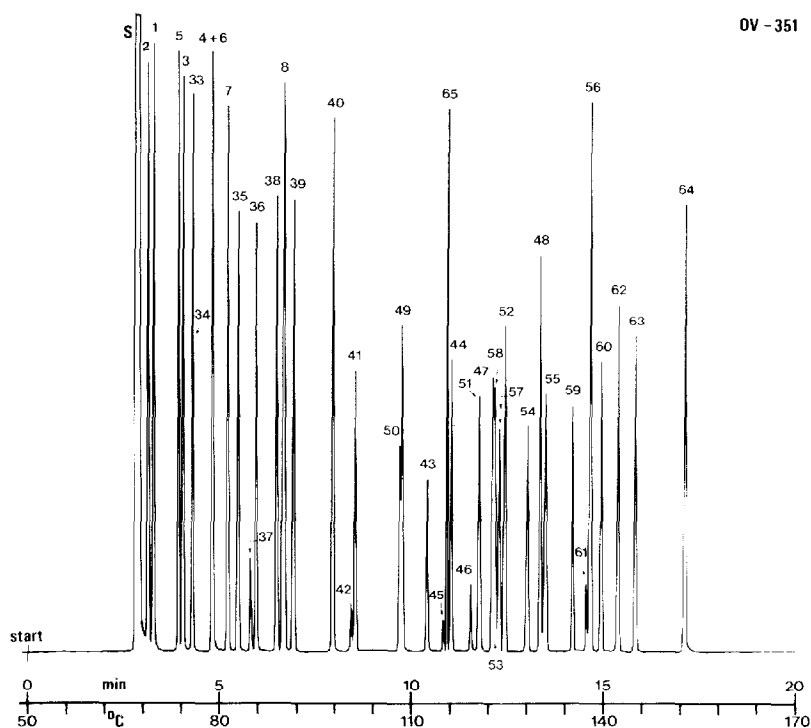


Fig. 5. Chromatogram of a mixture of branched-chain alcohols (1-8) and their butanoyl (33-40) and monochlorobutanoyl derivatives (41-64), analysed on OV-351. S = Solvent; compounds are identified in Table I.

(43); and finally, propyl 4-chlorobutanoate later than 1,1-dimethylpropyl 4-chlorobutanoate (61).

The relative retention times of the compounds studied, relative to *n*-tetradecane, varied on SE-30 between 0.12 and 0.86 and on OV-351 between 0.29 and 1.57 (Table I). By assigning the retention time relative to the corresponding alcohol, it can be seen that retention is maximized on SE-30 for 1,1-dimethylpropyl propanoate (13), 2-chloropropanoate (21) and butanoate (37), dimethylethyl 3-chloropropanoate (26) and monochlorinated dimethylethyl butanoates (42, 50 and 58). Retention is minimized for the parent methylethyl esters (9 and 33) and for all 3-methylbutyl monochloro esters (24, 32, 48, 56 and 64). As is evident from Table I, on OV-351 the retention is maximized for 1,1-dimethylpropyl esters (13, 21, 29, 37 and 45) and for dimethylethyl derivatives (50 and 58) and minimized for all 3-methylbutyl derivatives (16, 24, 32, 40, 48, 56 and 64). The last column in Table I shows that, like lower straight-chain alcohols^{13,14} greater retention times on OV-351 are observed for branched-chain alcohols. Methylethyl and 2-methylpropyl 3-chloropropanoates (25, 28) are, however, the only derivatives indicating the same trend. As is shown, 3-methyl-1-butanol (8), 1,1-dimethylpropyl esters (13, 21, 29 and 61) and dimethylethyl esters (34, 42 and 50) give rise to the greatest disparities between retention times on the columns used.

CONCLUSIONS

The present results show that branched-chain alcohols and their 2-chloropropanoyl, 3-chloropropanoyl, butanoyl, 2-chlorobutanoyl, 3-chlorobutanoyl and 4-chlorobutanoyl derivatives can be separated on SE-30, whereas on OV-351 incomplete separation is observed amongst the alcohols and their propanoyl and butanoyl derivatives. Complete separation of the individual components from the mixtures of alcohols and their C₃- and C₄-derivatives studied could not be achieved on any single column. The results show that alcohols and alkyl propanoates and also alcohols and alkyl butanoates are resolvable on SE-30, with one or several overlaps occurring for alkyl monochloro esters, particularly on OV-351. However, the mixtures can be separated by using columns with polar and non-polar stationary phases, although incomplete resolution occurred between some alkyl monochlorobutanoates.

ACKNOWLEDGEMENTS

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