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Capillary gas chromatography of higher alkylpolyoxyethylene glycols with an even number of carbon atoms in the alkyl group
Influence of type of derivatizing agent, alcohol chain length and oxyethylene chain length on the retention indices with a linear temperature increase

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Abstract

The influence of an increase in the length of the chain in the alkyl or polyoxyethylene glycol part of molecule of some homologous series of higher alkylpolyoxyethylene glycols and their derivatives on retention indices with a linear temperature increase was studied. The influence of the functional group supplied by the derivatizing agent on the retention index was also studied. Alkylpolyoxyethylene glycols with an even carbon number were converted by derivatization reactions into acetates, monochloroacetates, trifluoroacetates and trimethylsilyl ethers.

Keywords: Retention indices; Chain length; Derivatization, GC; Alkylpolyoxyethylene glycols; Glycols; Surfactants

1. Introduction

Alkylpolyoxyethylene glycols (APOEG) rank among important surfactants. They are not only produced industrially but also prepared in the laboratory by oxyethylenation of either the individual natural or synthesized aliphatic alcohols and their mixtures. The compounds are used mainly for the production of surfactants such as emulsifiers for the textile, paper and pharmaceutical industries, dispersants for acrylate paints and waxes, wetting, colouring and slashing agents for industry, and so on. Lower molecular mass APOEG have been used for the production of heat-carrying liquids.

The physical and chemical properties of these compounds are dependent on the alkyl and polyethylene glycol chain lengths. Hence great attention has been paid to the study of not only

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the physical parameters and chemical reactivities but also the gas chromatographic properties of APOEG. Papers dealing with these problems, including oxyethylenated derivatives of other series, were reviewed by Szymanowski [1], Voelkele [2] and others [3,4].

Papers dealing with both the identification of components in mixtures after their oxyethylenation and the determination of individual oxyethylenates and unreacted starting aliphatic alcohols [5–20] have demonstrated the great importance of controls for the efficient running of processes.

GC–MS with different ionization techniques has been used for identification of APOEG beside other components in more complex mixtures [21,22]. Separations have been carried out on analytical packed columns [5–19] and on capillary columns [20,22]. GC has been used in analyses for free underivatized APOEG [7,11,17,18,22] and derivatives of APOEG such as trimethylsilyl derivatives [6,9,10,12,14–16,20], methyl ethers [23] and acetates [5,8,13,15,16, 19,21].

Earlier work was reviewed by Evans and Haken [24]. Previous papers from our group [25–27] dealt with relationships between retention indices and the structures of halogenated and non-halogenated esters.

2. Experimental

2.1. Separated compounds

The following homologous series of higher alcohols with even numbers of carbon atoms were separated:

Non-derivatized compounds:

- higher aliphatic alcohols, $C_{12}OH$, $C_{14}OH$, $C_{16}OH$;
- *n*-alkylpolyoxyethylene glycols with the number (*n*) of oxyethylene glycol groups (EO = $-O-CH_2CH_2-$), *n*EO = 1–6, that is, $C_{12}EO_1OH$–$C_{12}EO_6OH$, $C_{14}EO_1OH$–$C_{14}EO_6OH$, and so on.

Derivatized compounds:

- trimethylsilyl derivatives (TMS) of
  higher aliphatic alcohols, $C_{12}TMS$, $C_{14}TMS$, $C_{16}TMS$;
  n-alkylpolyoxyethylene glycols with *n*EO = 1–6, that is, $C_{12}EO_1TMS$–$C_{12}EO_6TMS$, $C_{14}EO_1TMS$–$C_{14}EO_6TMS$, and so on;
- acetates (Ac) of
  higher aliphatic alcohols, $C_{12}Ac$, $C_{14}Ac$, $C_{16}Ac$;
  n-alkylpolyoxyethylene glycols with *n*EO = 1–6, that is $C_{12}EO_1Ac$–$C_{12}EO_6Ac$, $C_{14}EO_1Ac$–$C_{14}EO_6Ac$, and so on;
- trifluoroacetates (TFAc) of
  higher aliphatic alcohols, $C_{12}TFAc$, $C_{14}TFAc$, $C_{16}TFAc$;
  n-alkylpolyoxyethylene glycols with *n*EO = 1–6, that is, $C_{12}EO_1TFAc$–$C_{12}EO_6TFAc$, $C_{14}EO_1TFAc$–$C_{14}EO_6$ TFAc, and so on;
- monochloroacetates (MCAc) of
  higher aliphatic alcohols, $C_{12}MCAc$, $C_{14}MCAc$, $C_{16}MCAc$;
  n-alkylpolyoxyethylene glycols with the *n*EO = 1–5, that is, $C_{12}EO_1MCAc$–$C_{12}EO_5MCAc$, $C_{14}EO_1MCAc$–$C_{14}EO_5$ MCAc, and so on.

2.2. Preparation of derivatives

Trimethylsilyl derivatives were prepared by reaction of aliphatic alcohols or APOEG with BSA, acetates and trifluoroacetates by reaction with corresponding anhydrides, and monochloroacetates by reaction with monochloroacetyl chloride [28].

2.3. Apparatus

The studies were carried out on a Carlo Erba Fractovap Model 4160 gas chromatograph equipped with a flame ionization detector (Fisons Instruments, Milan, Italy), a CI-105 integrator and a TZ-4620 recorder (Laboratorní přístroje, Prague, Czech Republic). A laboratory-made glass capillary column of WCOT type (12 m × 0.7 mm I.D.) with OV-61-OH immobilized

stationary phase was used as a separation column.

2.4. Working conditions

The following conditions were used: column temperature, programmed linearly from 120 to 370°C at 5.5°C min^{-1}; injector and detector temperatures, 340°C; injection system, split, splitting ratio 1:15; and carrier gas, helium at a flow-rate of 1.4 ml min^{-1}.

Mixtures of the compounds studied were injected together with *n*-alkanes. First a mixture of a solution of APOEG in methanol and solution of their derivatives in benzene and then a solution of a mixture of *n*-alkanes in cyclohexane were drawn into a microsyringe.

3. Results and discussion

The retention indices (I_{PT}) were calculated according to following equation [29,30]:

$$I_{PTi} = 100z + 100n \cdot \frac{T_{Ri} - T_{Rz}}{T_{R(z+n)} - T_{Rz}}$$

where T_{Ri}, T_{Rz} and $T_{R(z+n)}$ are the retention temperatures of component i and *n*-alkanes eluting before and after this component, respectively, z and $z+n$ are the number of carbon atoms in *n*-alkanes eluting before and after component i, respectively, and $T_R = T_0 + rt_R$, T_0 being the starting temperature, r the temperature gradient and t_R the retention time.

The measurements were repeated six times and treated statistically. The average values of I_{PT} are given in Table 1. Then the increments of retention indices for the incoming EO group (ΔI_{EO}), incoming methylene groups (ΔI_{2CH_2}, ΔI_{4CH_2}) and incoming functional groups (ΔI_{3F}, ΔI_{Cl}, ΔI_{CF_3CO}, ΔI_{CH_3CO}, ΔI_{CH_2ClCO}, ΔI_{TMS}) were calculated according to the following equations and the values obtained are given in Tables 2–4:

In Table 2:

$$\Delta I_{EO} = I_{nEO} - I_{(n-1)EO}$$

In Table 3:

Table 1
Retention indices, I_{PT}, of oxyethylenated higher aliphatic alcohols (C_{12}, C_{14}, C_{16}) and their acetates, monochloroacetates, trifluoroacetates and trimethylsilyl derivatives

Compounds	*n*EO	C_{12}	C_{14}	C_{16}
Alcohols	0	1554.5	1754.9	1961.6
	1	1806.4	2015.8	2225.4
	2	2133.2	2332.8	2558.4
	3	2445.1	2644.1	2856.6
	4	2764.1	2947.1	3167.8
	5	3074.2	3252.0	3477.1
	6	3380.1		
Acetates	0	1674.7	1878.2	2081.2
	1	1978.9	2176.9	2383.9
	2	2282.3	2481.8	2683.7
	3	2582.6	2788.6	2990.9
	4	2897.0	3089.8	3297.2
	5	3212.5	3397.4	3605.9
	6	3506.8	3693.2	3900.0
Monochloroacetates	0	1943.8	2139.1	2351.8
	1	2242.8	2444.3	2652.1
	2	2553.8	2751.5	2960.4
	3	2868.8	3061.0	3267.8
	4	3170.2	3370.0	3580.2
	5	3481.4	3680.5	3890.0
Trifluoroacetates	0	1455.6	1644.6	1842.1
	1	1743.7	1938.2	2131.0
	2	2027.8	2222.1	2412.4
	3	2317.2	2509.6	2696.0
	4	2600.9	2789.0	2980.6
	5	2890.2	3080.2	3269.6
	6	3173.3	3371.9	3542.4
Trimethylsilyl ethers	0	1571.6	1765.6	1967.8
	1	1874.2	2070.0	2266.8
	2	2172.7	2370.3	2565.8
	3	2472.0	2666.1	2868.0
	4	2774.4	2964.2	3160.0
	5	3065.5	3257.1	3460.0
	6	3366.7	3555.2	3760.0

$$\Delta I_{2CH_2} = I_{C_{16}} - I_{C_{14}};\ I_{C_{14}} - I_{C_{12}}$$

$$\Delta I_{4CH_2} = I_{C_{16}} - I_{C_{12}}$$

In Table 4:

$$\Delta I_{3F} = I_{Ac} - I_{TFAc}$$

$$\Delta I_{Cl} = I_{MCAc} - I_{Ac}$$

$$\Delta I_{CF_3CO} = I_{APOEG} - I_{TFAc}$$

Table 2
Increments of retention index, ΔI_{EO}, for an incoming oxyethylene group into alkylpolyoxyethylene glycol molecule

Compounds	nEO	C_{12}	C_{14}	C_{16}
Alcohols	1	251.9	260.9	263.8
	2	326.8	317.0	333.0
	3	311.9	311.3	298.2
	4	319.0	303.0	311.2
	5	310.0	304.9	309.3
	6	305.9		
Acetates	1	304.2	298.7	302.7
	2	303.4	304.9	299.8
	3	300.3	306.8	307.2
	4	314.4	301.2	306.3
	5	315.5	307.6	308.7
	6	294.3	295.8	294.1
	7	308.9		
Monochloroacetates	1	299.0	305.2	300.3
	2	311.0	307.2	308.3
	3	315.0	309.5	307.8
	4	301.4	309.0	312.3
	5	311.2	310.5	309.8
	6	309.0		
Trifluoroacetates	1	288.1	293.6	288.9
	2	284.1	283.9	281.4
	3	289.4	287.5	283.6
	4	283.7	279.4	284.6
	5	289.3	291.9	289.0
	6	283.7	291.7	
Trimethylsilyl ethers	1	302.6	304.4	299.0
	2	298.5	300.3	299.0
	3	299.3	295.8	302.2
	4	302.4	298.1	292.0
	5	291.1	292.9	300.0
	6	301.2	298.1	300.0
	7	299.1	302.8	
	8	303.7		

$$\Delta I_{CH_3CO} = I_{Ac} - I_{APOEG}$$

$$\Delta I_{CH_2ClCO} = I_{MCAc} - I_{APOEG}$$

$$\Delta I_{TMS} = I_{TMS} - I_{APOEG}$$

Some information about influence of the incoming groups on the retention indices is evident from Fig. 1, where the dependence of retention indices on the number of oxyethylene groups is shown.

Table 3
Increments of retention index, ΔI_{2CH_2}, and ΔI_{4CH_2}, for two and four methylene groups, respectively, in the increasing aliphatic chain of polyoxyethylene glycol molecules

Compounds	nEO	$C_{16} - C_{14}$	$C_{14} - C_{12}$	$C_{16} - C_{12}$
Alcohols	0	206.7	200.4	407.1
	1	209.6	209.4	419.0
	2	225.3	199.6	425.2
	3	212.5	199.0	411.5
	4	220.7	183.0	403.7
	5	225.1	177.8	402.9
Acetates	0	203.0	203.5	406.5
	1	207.0	198.0	405.0
	2	201.9	199.5	401.4
	3	202.3	206.0	408.3
	4	207.4	192.8	400.2
	5	208.5	184.9	393.4
	6	206.8	186.4	393.2
Monochloro-acetates	0	212.7	195.3	408.0
	1	207.8	201.5	409.3
	2	208.9	197.7	406.6
	3	206.8	192.2	399.0
	4	210.2	199.8	410.0
	5	209.5	199.1	408.6
Trifluoro-acetates	0	197.5	189.0	386.5
	1	192.8	194.5	387.3
	2	190.3	194.3	384.6
	3	186.4	192.4	378.8
	4	191.6	188.1	379.7
	5	189.4	190.0	379.4
	6	170.5	198.6	369.1
Trimethylsilyl ethers	0	202.2	194.0	396.2
	1	196.8	195.8	392.6
	2	195.5	197.6	393.1
	3	201.9	194.0	396.0
	4	195.8	189.8	385.6
	5	202.9	191.6	394.5
	6	204.8	188.5	393.3

Fluctuations of some results occurred because the single homologous series of APOEG were not separated step by step but a mixture of three homologous series together with mixtures of the necessary *n*-alkanes was injected. Even if the products of oxyethylenation of aliphatic alcohols with both 3 and 6 mol of ethylene oxide were mixed in ratio of 1:1, a mixture with a uniform concentration of all components could not be

Table 4
Increments of retention index for incoming functional groups

Alcohol	nEO	ΔI_{3F}	ΔI_{Cl}	ΔI_{CF_3CO}	ΔI_{CH_3CO}	ΔI_{CH_2ClCO}	ΔI_{TMS}
C_{12}	0	219.1	269.1	98.9	120.2	389.3	17.1
	1	235.2	263.9	62.7	172.5	436.4	67.8
	2	254.5	271.5	105.4	149.1	420.6	39.5
	3	265.4	286.2	127.1	137.5	423.7	26.9
	4	296.1	273.2	163.2	132.9	406.1	10.3
	5	322.3	268.9	184.0	138.3	407.2	−8.7
	6	333.5		206.8			−13.4
C_{14}	0	233.6	260.9	110.3	123.3	384.2	10.7
	1	238.7	267.4	77.6	161.1	428.5	54.2
	2	259.7	269.7	110.7	149.0	418.7	37.5
	3	279.0	272.4	134.5	144.5	416.9	22.0
	4	300.8	280.2	158.1	142.7	422.9	17.1
	5	317.2	283.1	171.8	145.4	428.5	5.1
	6	321.3					
C_{16}	0	239.1	270.6	119.5	119.6	390.2	6.2
	1	252.9	268.2	94.4	158.5	426.7	41.4
	2	271.3	276.7	146.0	125.3	402.0	7.4
	3	294.9	276.9	160.6	134.3	411.2	11.4
	4	316.6	283.0	187.2	129.4	412.4	−7.8
	5	336.3	284.1	207.5	128.8	412.9	−17.1
	6	357.6					

prepared. Ethoxylates of C_{12} aliphatic alcohol were found in the prepared mixture to the greatest extent and ethoxylates of C_{14} alcohol the least. Ethoxylates with the 1–6 EO groups were present in the mixture to the greatest extent. Trace amounts of ethoxylates of C_{10} and C_{18} alcohols present in C_{12}–C_{16} alcohols were not evaluated, nor were the small amounts of ethoxylates with nEO > 6.

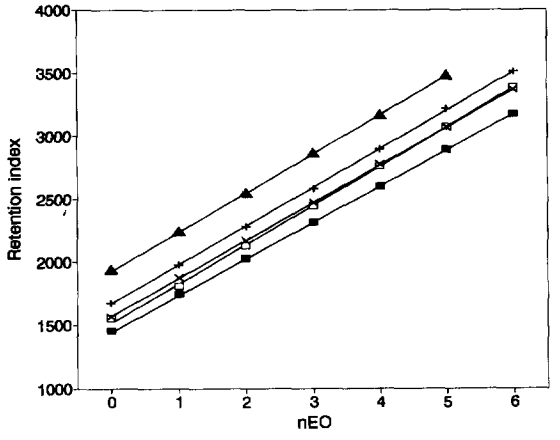

Fig. 1. Dependence of retention indices of derivatives of oxyethylated lauryl alcohol on oxyethylene group number. □ = Alcohols; + = acetates; ▲ = monochloroacetates; ■ = trifluoroacetates; × = trimethylsilyl ethers.

This work was aimed at studying the retention indices of APOEG and their derivatives with nEO < 6 with a linear temperature increase and the dependence of the retention indices on the structures of the molecules. The laboratory-prepared mixtures should be similar to the industrial products with a lower degree of ethoxylation.

As is evident from Table 1 and the chromatogram in Fig. 2, both free hydroxy compounds and their derivatives eluted in the following order: C_{12}, C_{14}, $C_{12}EO_1$, C_{16}, $C_{14}EO_1$, $C_{12}EO_2$, $C_{16}EO_1$, $C_{14}EO_2$, $C_{12}EO_3$, $C_{16}EO_2$, $C_{14}EO_3$, and so on. The order of elution of the separated compounds is in accordance with their increasing molecular mass.

Analogously to the case of the analysis of higher aliphatic alcohols, derivatization of oxyethylenates prevented the formation of hydrogen

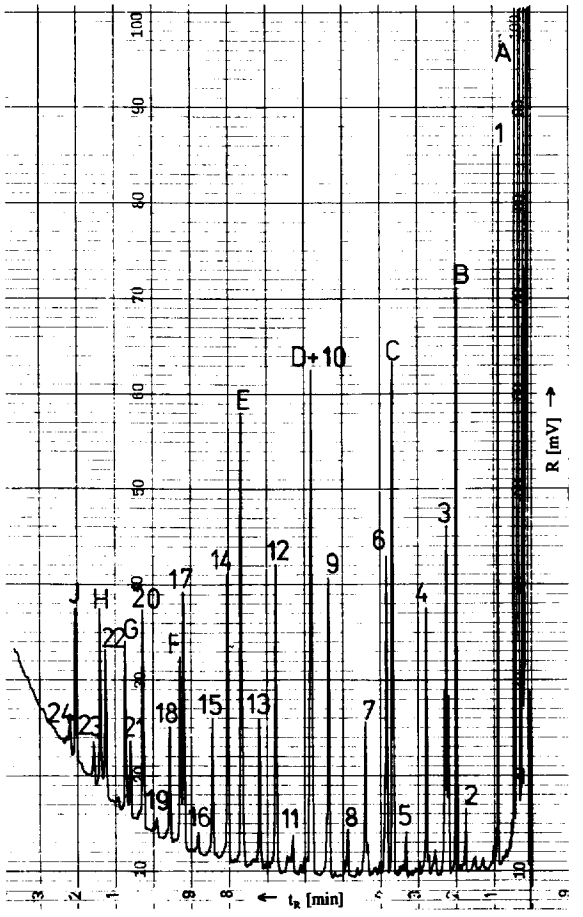

Fig. 2. Chromatogram of the separation of alkylpolyoxyethylene glycol trifluoroacetates. 1 = C_{12}TFAc; 2 = C_{14}TFAc; 4 = C_{16}TFAc; 3, 6, 9, 12, 15, 18, 21, 24 = $C_{12}EO_1$TFAc to $C_{12}EO_8$TFAc; 5, 8, 11, 14, 17, 20, 23 = $C_{14}EO_1$TFAc to $C_{14}EO_7$TFAc; 7, 10, 13, 16, 19, 22 = $C_{16}EO_1$TFAc to $C_{16}EO_6$TFAc; A, B, C, D, E, F, G, H, J = C_{13}, C_{17}, C_{20}, C_{24}, C_{28}, C_{32}, C_{38}, C_{40} *n*-alkanes, respectively.

bonds and the separation was considerably improved.

The methods of derivatization used, i.e., acylation and silylation, always resulted in an increase in the molecular mass of APOEG corresponding to the mass of the incoming functional group, e.g., CH_3CO–, 43.03; $(CH_3)_3Si$–, 73.21; $ClCH_2CO$–, 77.48; and CF_3CO, 97.02.

Although the introduction of a trifluoroacetyl group into a molecule of APOEG increases the relative molecular mass of the analyte compounds the most, the interaction between the separated derivatives and the stationary phase is strongly affected by the presence of three fluorine atoms. This resulted in a decrease in the retention indices of TFAc in comparison with non-derivatized APOEG (see Table 1). The decrease in the retention indices is evident from the values for ΔI_{CF3CO} in Table 4. These increments are always increased for all three homologous series of trifluoroacetates in order from derivatives with nEO = 1 to those with nEO = 5. The chromatogram of the separation of trifluoroacetates of APOEG is shown in Fig. 2.

Analogous results were obtained for ΔI_{3F} values that were calculated as the differences between the retention indices of acetates and the corresponding trifluoroacetates. These values pointed not only to the influence of three incoming fluorine atoms into the molecule of acetates on their interaction with the stationary phase but also at the possibility of shortening the analysis time of APOEG after their conversion into TFAc. The influence of incoming nEO on ΔI_{3F} values is evident from Table 4; the latter values increase with increasing nEO.

If APOEG are converted into acetates, the possibility of hydrogen bonding with the stationary phase is limited even though the total molecular mass of the derivatives is always increased by 42.03. This value is nearly identical with the relative molecular mass corresponding to three methylene groups (3 × 14.02), but the increase in retention indices in comparison with non-acetylated APOEG fluctuates from 128.8 to 172.5 retention units.

The increments in retention indices for an incoming acetyl group, ΔI_{CH_3CO}, decrease gradually with increasing number of oxyethylene groups in a molecule of the ester for all three homologous series (see Table 4). The largest decreases in ΔI_{CH_3CO} values always occur for derivatives with lower nEO and very small values for derivatives with nEO = 4–6.

If we did not take into account the few fluctuating retention indices that occurred, we could draw analogous conclusions for the incoming monochloroacetyl group. Its entry into a molecule of APOEG resulted in a considerable

increase in retention times and large increases in the retention indices of the monochloracetate homologous series in comparison with both the non-derivatized APOEG (436–402 units) and acetates (263–284 units) (see Tables 1 and 4).

The ΔI_{Cl} increments for the chlorine atom (see Table 4) were calculated as the difference between the retention indices of monochloroacetates and the corresponding acetates. They characterize the influence of a chlorine atom on an interaction between separated monochloroacetates and the stationary phase. The ΔI_{Cl} values increase only moderately, by a few retention index units, with increase in *n*EO.

The analysis of APOEG after their conversion into trimethylsilyl derivatives is very interesting. The incoming trimethylsilyl group results in only a small increase in the retention indices, by 5–67 units in comparison with the retention indices of non-derivatized APOEG (see Tables 1 and 4). Even a change in the elution order occurs for some derivatives such as $C_{12}EO_5TMS$, $C_{12}EO_6TMS$, $C_{16}EO_4TMS$ and $C_{16}EO_5TMS$. These derivatives elute before non-derivatized APOEG. The ΔI_{TMS} increment values decrease step by step with increasing *n*EO in molecule of derivatives (see Table 4). The suitability of the conversion of both the higher aliphatic alcohols and their oxyethylates into trimethylsilyl derivatives or trifluoracetates is evident from Table 4.

Retention index increments for the incoming oxyethylene group ΔI_{EO} are balanced and fluctuate only moderately by a few retention index units for all the derivatized forms (see Table 2). The ΔI_{EO} values move above a value of 300 for nearly all acetates and monochloroacetates, around 300 for trimethylsilyl derivatives and below 290 for trifluoroacetates. The largest ΔI_{EO} values are for the second EO group and the smallest for the first EO group.

The influence of an increase in the alkyl chain length on the I_{PT} values was studied by means of retention index increments for two and four incoming methylene groups (see Table 3). The values of the increments for homologous series of free APOEG, their acetates and monochloroacetates for two CH_2 groups vary close to a value of 200 and for four CH_2 groups around 400, or they are moderately above these values. The ΔI_{2CH_2} and ΔI_{4CH_2} values for trifluoroacetates are always below 200 and 400, respectively, fluctuating around values of 190 and 380, respectively. The results for trimethylsilyl derivatives are interesting. In that case the ΔI_{2CH_2} values for two incoming CH_2 groups into a C_{14} alkyl group fluctuate around 200 but ΔI_{2CH_2} for two incoming CH_2 groups into a C_{12} alkyl group fluctuate around 194. In a similar way for ΔI_{4CH_2}, the values for four incoming CH_2 groups into a C_{12} alkyl group are below 400, fluctuating around a value of 394.

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