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## FATTY ACIDS

### XI\*. THE SYNTHESIS OF EPOXIDES FROM METHYL OCTADECADIENOATES AND A STUDY OF THEIR CHROMATOGRAPHIC PROPERTIES

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#### SUMMARY

Tetrahydrofuranyl and tetrahydropyranyl epoxides of the isomers of methyl octadecanoate have been prepared by the oxymercuration–demercuration reaction of dimethylene-interrupted methyl *cis,cis*-octadecadienoates. The thin-layer chromatography (on silica gel and silical gel impregnated with silver nitrate) and gas-liquid chromatography (on OV-101 and Silar 10C phases) of the derivatives have been studied. The mass spectra of these compounds are also reported.

#### INTRODUCTION

Oxymercuration–demercuration reactions of dienes and unsaturated alcohols to yield diols, tetrahydrofurans and tetrahydropyrans are well documented<sup>2,3</sup>. Gunstone and Inglis<sup>4</sup> have extended this reaction to enable the rapid identification of certain unsaturated long-chain alcohols. In an earlier study we reported the result of the oxymercuration–demercuration reactions of methyl undecenoates, undecynoates and some diunsaturated C<sub>18</sub> fatty esters<sup>5</sup>.

In order to further our study of the chemical properties of the dimethylene-interrupted methyl *cis,cis*-octadecadienoates<sup>6</sup>, we have prepared all of the positional isomers of the tetrahydrofuranyl (THF) (1), and all except one of the tetrahydropyranyl (THP) (2), derivatives from this unique series of diunsaturated fatty esters.



\* For Part X, see ref. 1.

Our studies are concerned with the preparative and analytical value of such oxyheterocycles.

## EXPERIMENTAL

### *General method for the oxymercuration-demercuration of methyl octadecadienoates*

A mixture of the methyl octadecadienoate (40 mg), mercuric acetate (100 mg), tetrahydrofuran (4 ml) and water (6 ml) was shaken for 4 days. Sodium tetrahydroborate (40 mg) in aqueous sodium hydroxide (3 M, 10 ml) was then added. The reaction mixture was saturated with sodium chloride and extracted with diethyl ether. The ether extract gave a mixture of the tetrahydrofuranyl and tetrahydropyranyl derivatives (38 mg). Separation of the tetrahydrofuranyl ( $R_F = 0.4$ ) from the tetrahydropyranyl ester ( $R_F = 0.5$ ) was achieved by preparative TLC on silical gel, using diethyl ether-light petroleum (b.p. 60–80°) (1:9) as developing solvent. The results of the oxymercuration-demercuration reactions of all of the dimethylene-interrupted methyl *cis,cis*-octadecadienoates are summarized in Table I.

### *Spectroscopic and chromatographic procedures*

IR spectra were recorded on a Perkin-Elmer 337 grating spectrophotometer using neat liquids sandwiched between 5-mm potassium bromide plates. Mass spectra were recorded on a Hitachi RMS-4 mass spectrometer. Samples were introduced by direct insertion. Spectra were normally run at 70 eV with a source pressure of  $10^{-6}$  Torr and a temperature of 150–180°. NMR spectra were recorded on 15% solutions in deuterated chloroform using a Hitachi-Perkin-Elmer 20A spectrometer (60 MHz). Chemical shifts were measured in ppm downfield from internal tetramethylsilane ( $\tau = 10$ ).

Analytical (on 0.3-mm plates) and preparative TLC (on 0.7-mm plates) were carried out in the usual manner using diethyl ether-light petroleum (b.p. 60–80°) as developer. GLC analyses were performed on a Pye 104 or a Varian 940 chromatograph and retention values are reported as equivalent chain lengths<sup>7</sup>.

## RESULTS AND DISCUSSION

### *Synthesis*

Cyclization of the methyl *cis,cis*-octadecadienoates to the THF and THP derivatives was effected by treating the substrate with mercuric acetate in a mixture of water and tetrahydrofuran. The results are summarized in Table I and we can make the following additional comments.

The reaction product also contained a mixture of methyl dihydroxyoctadecanoate isomers, but we made no special effort to isolate and further identify these compounds. However, we noted their polar character in thin-layer chromatography (TLC) and the absorption  $\nu$  (O–H) of these compounds in the IR spectrum (3400  $\text{cm}^{-1}$ ).

Separation of the THF and THP derivatives was readily achieved by preparative TLC on silica, since the five-membered cyclic compound is more polar than the six-membered cyclic derivative. The gas-liquid chromatographic (GLC) analysis on Silar 10C of the isolated TLC fraction of the THF derivative indicates the presence of

TABLE I

RESULTS OF THE OXYMERCURATION-DEMERCURATION REACTIONS OF METHYL OCTADECADIENOATES

Substrate isomer (mg)	Hg(OAc) <sub>2</sub> (mg)	H <sub>2</sub> O-THF (3:2) (ml)	Reaction products*		
			Recovered (unchanged) C <sub>18:2</sub> (%)	THF ester (%)	THP ester (%)
$\Delta^{2c,6c}$ (55)	120	12	15	19	9
$\Delta^{3c,7c}$ (35)	86	9	20	34	27
$\Delta^{4c,8c}$ (33)	82	8	4	51	16
$\Delta^{5c,9c}$ (40)	119	10	—	45	24
$\Delta^{6c,10c}$ (61)	176	15	—	56	26
$\Delta^{7c,11c}$ (64)	134	16	4	47	23
$\Delta^{8c,12c}$ (90)	126	22	58	27	8
$\Delta^{9c,13c}$ (50)	104	12	34	30	12
$\Delta^{10c,14c}$ (50)	99	12	—	73	20
$\Delta^{11c,15c}$ (62)	134	15	9	52	22
$\Delta^{12c,16c}$ (33)	76	9	26	54	9
$\Delta^{13c,17c}$ (29)	84	8	11	—	56
$\Delta^{7r,11r}$ (14)	56	5	12	48	40
$\Delta^{8r,12r}$ (16)	36	5	13	50	37
$\Delta^{5c,10c}$ (35)	107	10	—	30	—

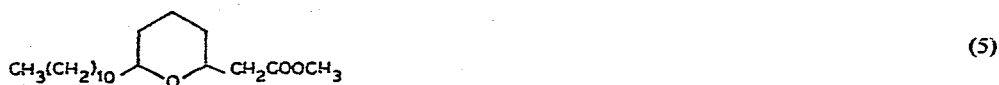
\* Values obtained by preparative TLC.

two components, which we believe to be a mixture of the *E* and *Z* isomers (3,4), as the mass spectra of the sub-fractions (isolated by preparative GLC) were identical.



The GLC analysis of the TLC-isolated THP derivative gave only a single peak on Silar 10C.

In the formation of the five-membered cyclic derivatives only one positional isomer is formed. Two THP positional isomers are possible from each substrate isomer, except in the case of the  $\Delta^{2c,6c}$  ester where only one six-membered cyclic derivative (5) is obtained. The  $\Delta^{13c,17c}$  ester does not give a six-membered heterocycle.



All of the above observations can be explained mechanistically, assuming that one olefinic bond is first hydrated, followed by the formation of a mercury complex

TABLE II

EQUIVALENT CHAIN LENGTHS OF MIXTURES OF THE *E* AND *Z* ISOMERS OF THE THF EPOXIDES OF METHYL OCTADECANOATE ON SILAR 10C AND OV-101

Substrate isomer C <sub>18:2</sub>	Epoxide		ECL on Silar 10C*		ECL on OV-101**
	y	x	<i>E</i> isomer	<i>Z</i> isomer	
Δ <sup>2c,6c</sup>	1	11	21.44 (30)	21.70 (70)	18.65
Δ <sup>3c,7c</sup>	2	10	21.29 (45)	21.53 (55)	18.54
Δ <sup>4c,8c</sup>	3	9	21.27 (49)	21.61 (51)	18.53
Δ <sup>5c,9c</sup>	4	8	21.34 (37)	21.71 (63)	18.55
Δ <sup>6c,10c</sup>	5	7	21.36 (48)	21.74 (52)	18.56
Δ <sup>7c,11c</sup>	6	6	21.40 (47)	21.76 (53)	18.55
Δ <sup>8c,12c</sup>	7	5	21.49 (42)	21.84 (58)	18.56
Δ <sup>9c,13c</sup>	8	4	21.58 (36)	21.92 (64)	18.59
Δ <sup>10c,14c</sup>	9	3	21.65 (42)	21.98 (58)	18.62
Δ <sup>11c,15c</sup>	10	2	21.80 (41)	22.14 (59)	18.71
Δ <sup>12c,16c</sup>	11	1	21.97 (57)	22.30 (43)	18.83
Δ <sup>13c,17c</sup>	12	0	22.45 (44)	22.82 (56)	18.85
Δ <sup>7r,11r</sup>	6	6		21.52	18.39
Δ <sup>8r,12r</sup>	7	5		21.66	18.40
Δ <sup>5c,10c</sup> ***					

\* 10% Silar 10C on Chromosorb W at 180° with nitrogen as carrier gas. The relative area percentage of the components is indicated in parentheses.

\*\* 1.5% OV-101 on Chromosorb W at 180° with nitrogen as carrier gas.

\*\*\* THF epoxide is not formed.

TABLE III

EQUIVALENT CHAIN LENGTHS OF MIXTURES OF POSITIONAL ISOMERS OF THE THP EPOXIDES OF METHYL OCTADECANOATE ON SILAR 10C AND OV-101

Conditions as in Table II.

Substrate isomer C <sub>18:2</sub>	Epoxide		ECL		Epoxide		ECL	
	m	n	Silar 10C	OV-101	m	n	Silar 10C	OV-101
Δ <sup>2c,6c</sup>	0	11	—	—	1	10	19.72	18.02
Δ <sup>3c,7c</sup>	1	10	19.62	17.94	2	9	19.62	17.94
Δ <sup>4c,8c</sup>	2	9	19.62	17.84	3	8	19.62	17.84
Δ <sup>5c,9c</sup>	3	8	19.65	17.85	4	7	19.65	17.85
Δ <sup>6c,10c</sup>	4	7	19.69	17.85	5	6	19.69	17.85
Δ <sup>7c,11c</sup>	5	6	19.74	17.89	6	5	19.74	17.89
Δ <sup>8c,12c</sup>	6	5	19.86	17.93	7	4	19.86	17.93
Δ <sup>9c,13c</sup>	7	4	20.03	17.98	8	3	20.03	17.98
Δ <sup>10c,14c</sup>	8	3	20.06*	18.08	9	2	20.21*	18.08
Δ <sup>11c,15c</sup>	9	2	20.28**	18.14*	10	1	20.64**	18.40*
Δ <sup>12c,16c</sup>	10	1	20.60***	18.33*	11	0	21.19***	18.59*
Δ <sup>13c,17c</sup> ‡								
Δ <sup>7r,11r</sup>	5	6	19.73	17.88	6	5	19.73	17.88
Δ <sup>8r,12r</sup>	6	5	19.81	17.94	7	4	19.81	17.94
Δ <sup>5c,10c</sup>	3	8	19.66	17.86	4	7	19.66	17.86

\* Shoulder separation.

\*\* Twin-peak separation.

\*\*\* Baseline separation.

‡ THP epoxide is not formed.

with the remaining olefinic bond. Cyclization is then achieved by the attack of the lone pair of the oxygen of the hydroxyl group on the carbon atoms of the remaining double bond which is complexed to mercuric acetate.

Similar reactions were carried out on  $C_{18:2}$  (7*t*,11*t*), (8*t*,12*t*) and (5*c*,10*c*) isomers. The results and physical properties are included in Tables I-III.

### Chromatography

**Thin-layer chromatography.** The THF and THP derivatives were readily separated on silica layers (E. Merck; silica gel GF 254, Type 60) and even better separations were obtained on thin layers of silica gel impregnated with 10% silver nitrate. The TLC of these two series of compounds, as mixtures of the *E* and *Z* isomers or of the positional isomers, conformed to a sinusoidal pattern on TLC plates (see Figs. 1 and 3). This sinusoidal behaviour of the positional isomers of unsaturated fatty esters in silver-ion TLC and of methyl hydroxyoctadecanoate isomers on silica gel layers was first reported by Morris and Wharry<sup>8</sup> and confirmed by Gunstone *et al.*<sup>9</sup>.

On silica gel layers (Fig. 1) the THP derivatives gave an average  $R_F$  value of 0.53, while the THF derivatives exhibited a sinusoidal pattern with a minimum  $R_F$  value (0.35) due to the derivative derived from the  $\Delta^{4c,8c}$  isomer and a maximum  $R_F$  value (0.49) for the derivative from the  $\Delta^{10c,14c}$  isomer. As the  $\Delta^{13c,17c}$  isomer does not

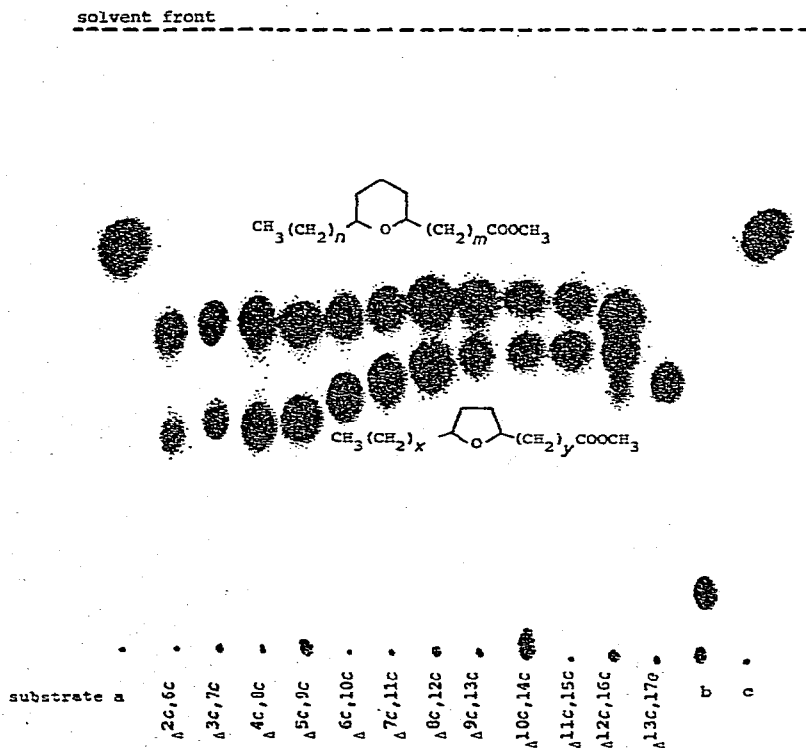


Fig. 1. TLC of the isomers of epoxides of methyl octadecanoates on silica gel. Developer, diethyl ether-light petroleum (2:11). Substrate: a = methyl stearate; b = methyl 5(6)-hydroxy-*cis*-10-octadecenoate; c = methyl *cis,cis*-5,9-octadecadienoate.

yield a THP derivative, only one component (the THF derivative) was available from this substrate. Methyl stearate ( $R_F = 0.65$ ), methyl *cis,cis*-5,9-octadecadienoate ( $R_F = 0.67$ ) and methyl 5(6)-hydroxy-*cis*-10-octadecenoate ( $R_F = 0.10$ ) were used as reference standards. The THP and THF derivatives were slightly more polar than the saturated or unsaturated fatty esters, but less polar than a hydroxy fatty ester, in TLC.

As only one positional isomer of the THF derivative was produced from each fatty ester substrate, we were able to demonstrate the sub-fractionation of positional THF isomers in TLC on silica gel by employing the derivatives obtained from the  $\Delta^{4c,8c}$ ,  $\Delta^{5c,9c}$ ,  $\Delta^{6c,10c}$  and  $\Delta^{7c,11c}$  isomers [*i.e.*, the methyl 5,8- ( $R_F = 0.28$ ); 6,9- ( $R_F = 0.30$ ); 7,10- ( $R_F = 0.32$ ) and 8,11- ( $R_F = 0.34$ ) epoxyoctadecanoate respectively]. Complete separation into two distinct spots occurred when the difference in  $R_F$  values exceeded 0.04 (Fig. 2).

On silver nitrate-impregnated silica gel, the THF and THP derivatives conformed to a more pronounced sinusoidal curve and the separation of the two series of epoxyoctadecanoates was further enhanced (Fig. 3). The  $R_F$  values ranged from 0.64 to 0.72 for the THP derivatives and from 0.47 to 0.60 for the THF derivatives. Methyl *cis,cis*-5,9-octadecadienoate ( $R_F = 0.36$ ) exhibited a smaller  $R_F$  value than the epoxides as the  $\pi$ -electrons of the two olefinic bonds were complexed to the silver

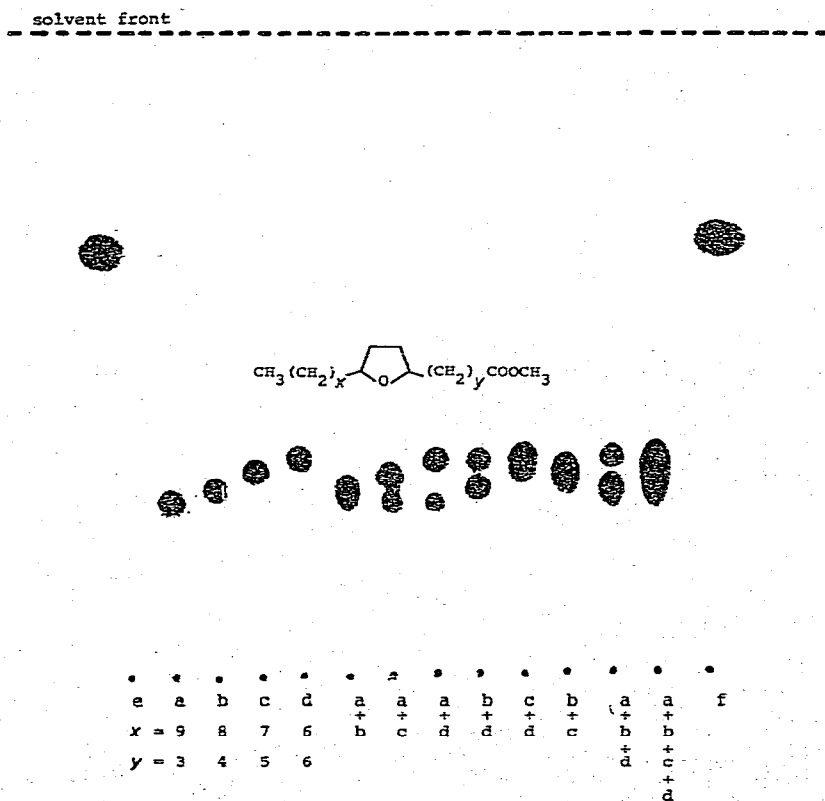


Fig. 2. TLC sub-fractionation of four positional THF isomers (a, b, c and d) on silica gel. Double development by diethyl ether-light petroleum (1:11). Substrate: e = methyl stearate; f = methyl *cis,cis*-5,9-octadecadienoate.

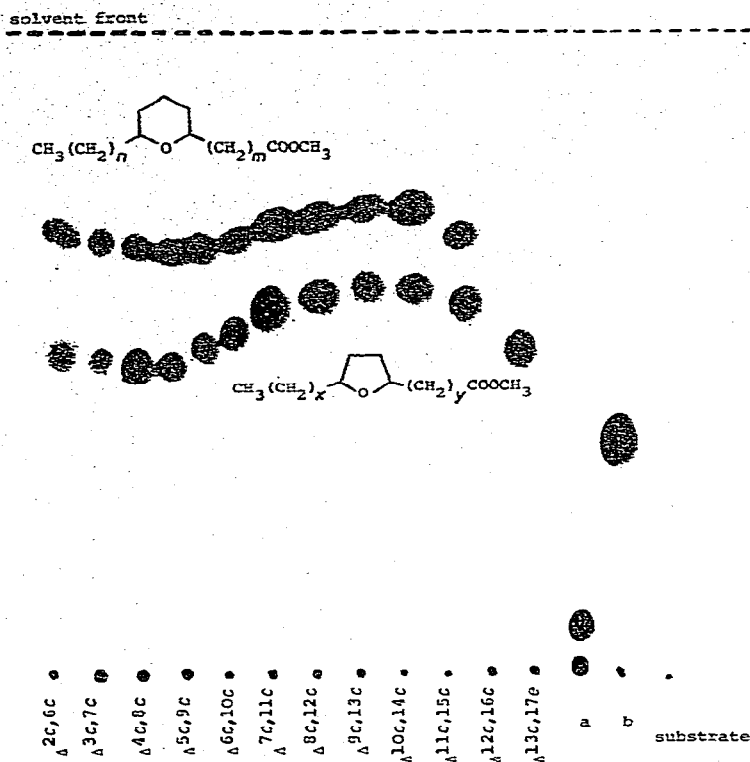


Fig. 3. TLC of the isomers of epoxides of methyl octadecanoates on 10% silver nitrate-impregnated silica gel. Developer, diethyl ether-light petroleum (2:11). Substrate: a = methyl 5(6)-hydroxy-*cis*-10-octadecenoate; b = methyl *cis,cis*-5,9-octadecadienoate.

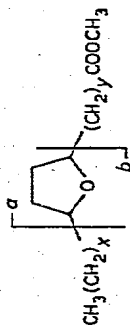
ions on the silica layers. Methyl 5(6)-hydroxy-*cis*-10-octadecenoate gave an  $R_F$  value of 0.07.

**Gas-liquid chromatography.** The GLC behaviour of both series of THF and THP derivatives is summarized in Tables II and III. The equivalent chain lengths (ECL) of these derivatives are presented in Fig. 4.

On the non-polar OV-101 stationary phase the THF derivatives gave higher ECL values than the THP compounds (18.54–18.85 and 17.84–18.59, respectively). The positional isomers of the THP derivatives obtained from the  $\Delta^{11c,15c}$  and  $\Delta^{12c,16c}$  substrates were separable on this phase as twin peaks.

On Silar 10C each THF derivative was further separated into two distinct peaks with an almost baseline separation. As the reaction of each substrate isomer ( $C_{18:2}$  fatty ester) gave only one positional THF derivative, we believe that each sub-fraction corresponds to the *E* and *Z* isomers, as these fractions, when isolated by preparative GLC, exhibited identical mass spectra. We have arbitrarily assigned the less polar THF derivative as the *E* isomer and the more polar component as the *Z* isomer. This assignment follows the usual behaviour of the *E* and *Z* isomers of the methyl octadecenoates on polar stationary phases. The ECL values of the *E* isomers of the THF derivatives ranged from 21.53 to 22.82, and those of the *Z* isomers were in the range 21.27–22.45. The THF derivatives obtained from the  $C_{18:2}$  (7*t*,11*t*) and (8*t*,12*t*) sub-

TABLE IV  
 MASS SPECTRA (70 eV) OF MIXTURES OF THE GEOMETRIC ISOMERS OF THE THF EPOXIDES OF METHYL OCTADECANOATES  
 Values quoted are  $m/e$ , origin of fragment and intensity relative to the base peak = 100.



Substrate	Epoxide	$M+1$	$M$	$a$	$a-32$	$b$	Others			
$C_{18:2}$	$x$	$y$								
$\Delta^{15,16c}$	11	1	313 (100)	312 (22)	143 (80)	111 (58)	239 (46)	311 ( $M-1,22$ )	294 ( $M-18,13$ )	125 ( $a-18,44$ )
$\Delta^{15,17c}$	10	2	313 (25)	312 (3)	157 (86)	125 (39)	225 (11)	311 ( $M-1,6$ )	207 ( $b-18,4$ )	55 (100)
$\Delta^{14,18c}$	9	3	313 (100)	312 (5)	171 (58)	139 (34)	211 (46)	311 ( $M-1,9$ )	153 ( $a-18,14$ )	121 ( $a-50,13$ )
$\Delta^{13,19c}$	8	4	313 (100)	312 (42)	185 (56)	153 (39)	197 (33)	311 ( $M-1,20$ )	167 ( $a-18,27$ )	135 ( $a-50,30$ )
$\Delta^{12,20c}$	7	5	313 (100)	312 (10)	199 (21)	167 (16)	183 (22)	311 ( $M-1,23$ )	149 ( $a-50,17$ )	
$\Delta^{11,21c}$	6	6	313 (7)	312 (13)	213 (30)	181 (27)	169 (50)	311 ( $M-1,48$ )	151 ( $b-18,27$ )	163 ( $a-50,21$ )
$\Delta^{10,22c}$	5	7	313 (41)	312 (5)	227 (100)	195 (65)	155 (97)	137 ( $b-18,76$ )	209 ( $a-18,45$ )	
$\Delta^{9,23c}$	4	8	313 (100)	312 (29)	241 (46)	209 (34)	141 (51)	123 ( $b-18,20$ )		
$\Delta^{10c,14c}$	3	9	313 (2)	312 (3)	255 (21)	223 (26)	127 (62)	109 ( $b-18,45$ )	57 (100)	
$\Delta^{11c,15c}$	2	10	313 (100)	312 (13)	269 (88)	237 (100)	113 (86)	95 ( $b-18,69$ )	219 ( $a-50,47$ )	
$\Delta^{12c,16c}$	1	11	313 (14)	312 (4)	283 (7)	251 (9)	99 (100)	81 ( $b-18,44$ )		
$\Delta^{13c,17c}$	0	12	313 (41)	312 (9)	297 (1)	263 (3)	85 (100)	67 ( $b-18,57$ )		



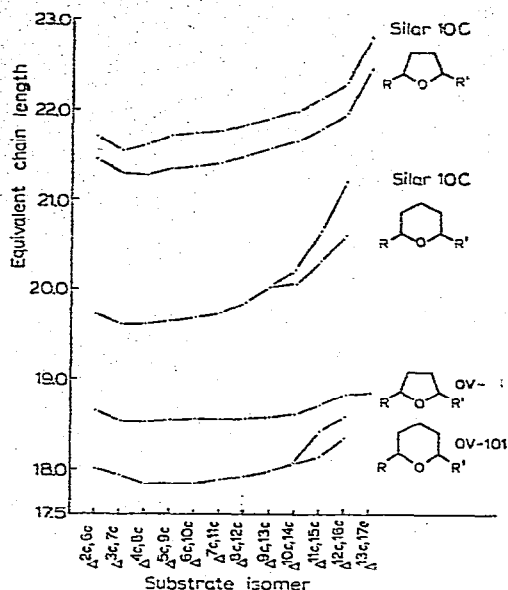


Fig. 4. Equivalent chain lengths of the THF and THP isomers.

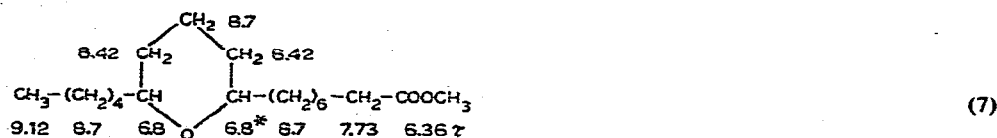
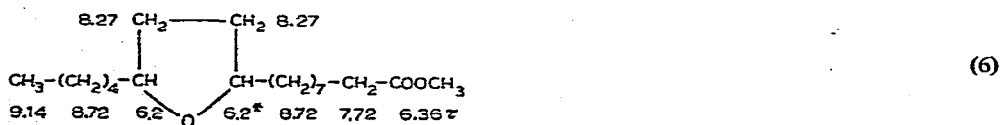
strates gave a single peak on Silar 10C with ECL values of 21.52 and 21.66 respectively. No further separation into *E* or *Z* isomers was observed. The ECL values of the same derivatives on OV-101 (18.39 and 18.40, respectively) were lower than those obtained by the reaction of the corresponding methyl *cis,cis*-octadecadienoates.

The ECL values of the THP derivatives ranged from 19.62 to 21.19 on Silar 10C. Within this series the positional THP derivatives obtained from the  $\Delta^{10c,14c}$ ,  $\Delta^{11c,15c}$  and  $\Delta^{12c,16c}$  substrate isomers were separable on this polar phase. The ECL values of the THP derivatives obtained from the  $C_{18:2}$  (*7t,11t*) and (*8t,12t*) isomers were almost identical to those obtained from the corresponding methyl *cis,cis*-octadecadienoates.

On both the polar and non-polar stationary phases the THP derivatives were less polar (*viz.* shorter retention times) than the THF derivatives.

#### Mass and NMR spectroscopy

The mass spectral data of the isolated fractions of the THF and THP derivatives are presented in Tables IV and V respectively.

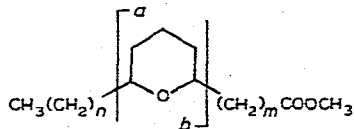


\* Broad signal.

TABLE V

## MASS SPECTRA OF MIXTURES OF THE POSITIONAL ISOMERS OF THE THP EPOXIDES OF METHYL OCTADECANOATES

Values quoted are  $m/e$ , origin of fragment, and intensity relative to the base peak = 100. Peaks are quoted in decreasing value of  $m/e$  except for mixtures where the fragments are separated into groups relating to each component of the mixture. Values marked with a star may result from more than one component of the mixture.



Substrate $C_{18:2}$	Epoxide		$M+1$	$M$	$a$	$b$	$a-32$	Others
	$n$	$m$						
$\Delta^{2c,6c}$	10	1	313 (100)	312 (35)	157 (38)	239 (24)	125 (62)	311 ( $M-1,30$ ) 221 ( $b-18,27$ )
$\Delta^{3c,7c}$	10	1	313 (100)	312 (32)	157 (15)	239 (7)	125 (20)	139* ( $a-18,44$ ) 311 ( $M-1,9$ ) 280 ( $M-32,16$ )
$\Delta^{4c,8c}$	9	2			171 (64)	225 (19)	139* (44)	153 ( $a-18,13$ )
	9	2	313 (100)	312 (9)	171 (16)	225 (1)	139 (11)	153* ( $a-18,24$ ) 311 ( $M-1,10$ )
$\Delta^{5c,9c}$	8	3			185 (24)	211 (5)	153* (24)	135 ( $a-50,12$ )
	8	3	313 (60)	312 (13)	185 (27)	211 (15)	153 (32)	167* ( $a-18,41$ ) 311 ( $M-1,20$ ) 39 (100)
$\Delta^{6c,10c}$	7	4			199 (23)	197 (40)	167* (41)	181 ( $a-18,30$ ) 149 ( $a-50,45$ )
	7	4	313 (100)	312 (12)	199 (11)	197 (16)	167 (13)	181* ( $a-18,17$ ) 149 ( $a-50,21$ ) 311 ( $M-1,10$ ) 295 ( $M-17,11$ )
$\Delta^{7c,11c}$	6	5			213 (10)	183 (21)	181* (17)	195 ( $a-18,18$ ) 163 ( $a-50,13$ )
	6	5	313 (100)	312 (12)	213 (12)	183 (28)	181 (16)	195* ( $a-18,30$ ) 311 ( $M-1,11$ )
$\Delta^{8c,12c}$	5	6			227 (11)	169 (16)	195* (30)	151 ( $b-18,22$ )
	5	6	313 (100)	312 (20)	227 (30)	169 (20)	195 (35)	209* ( $a-18,72$ ) 151 ( $b-18,24$ ) 311 ( $M-1,26$ ) 295 ( $M-17,20$ )
$\Delta^{9c,13c}$	4	7			241 (30)	155 (31)	209* (72)	223 ( $a-18,33$ ) 137 ( $b-18,60$ )
	4	7	313 (100)	312 (25)	241 (31)	155 (36)	209 (28)	223* ( $a-18,60$ ) 137 ( $b-18,28$ ) 311 ( $M-1,39$ ) 295 ( $M-17,43$ )
$\Delta^{10c,14c}$	3	8			255 (37)	141 (40)	223* (60)	237 ( $a-18,35$ ) 123 ( $b-18,25$ )
	3	8	313 (100)	312 (22)	255 (6)	141 (14)	223 (4)	237* ( $a-18,10$ ) 123 ( $b-18,12$ ) 311 ( $M-1,11$ ) 295 ( $M-17,11$ )
$\Delta^{11c,15c}$	2	9			269 (7)	127 (30)	237* (10)	109 ( $b-18,21$ )
	2	9	313 (33)	312 (22)	269 (10)	127 (43)	237 (15)	251* ( $a-18,47$ ) 109 ( $b-18,48$ )
$\Delta^{12c,16c}$	1	10			283 (23)	113 (47)	251* (47)	265 ( $a-18,23$ ) 95 ( $b-18,100$ )
	1	10	313 (14)	312 (9)	283 (2)	113 (30)	251 (3)	95 ( $b-18,34$ ) 265* ( $a-18,3$ ) 55 (100)
$\Delta^{13c,17c}$ *	0	11			297 (1)	99 (68)	265* (3)	81 ( $b-18,70$ )

\* THP epoxide is not formed.

The NMR spectra (60 MHz) of the THF and THP derivatives are represented by the isomers 6 and 7.

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