

## Note

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### Fatty acids

#### XVIII\*. Gas-liquid chromatographic behaviour of unsaturated furan esters

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(Received February 25th, 1977)

Unsaturated keto esters containing a furan system have been reported to induce smooth muscle contraction<sup>2</sup>. In the preparation of the entire series of isomeric C<sub>18</sub> furan esters<sup>3</sup>, we have made extensive use of the Wittig reaction to produce several unsaturated furan acid or ester derivatives using furfural as the starting material. As aprotic solvents (dimethyl sulphoxide or dimethylformamide) were employed in these preparations, large amounts (ca. 90%) of the *cis*-isomer were obtained with small amounts of the corresponding *trans*-compound. Gas-liquid chromatographic (GLC) separation and identification have so far been the best means of characterizing these intermediates.

The equivalent chain lengths (ECLs) of these compounds are summarized in Table I.

#### EXPERIMENTAL AND RESULTS

The GLC results were obtained under conditions similar to those described in Part XVII<sup>1</sup>.


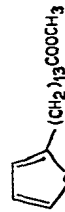
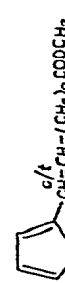
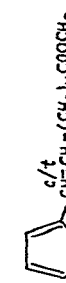


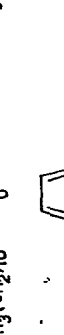
The ECLs of the C<sub>16</sub> and C<sub>18</sub> furan esters (compounds I and II) were included for comparison (see Table I). On polar stationary phases, compounds III and IV (both containing *cis*- and *trans*-isomers) were well separated into two distinct peaks. The major component was regarded as the *cis*-isomer and the minor component the corresponding *trans*-compound. This assumption was based on the experience that *cis*-ethylenic C<sub>18</sub> esters with the unsaturated centre between positions C<sub>2</sub> and C<sub>3</sub> gave lower ECLs than the corresponding *trans*-ethylenic C<sub>18</sub> esters on polar stationary phases<sup>4-6</sup>. Further, catalytic hydrogenation of such mixtures in all instances gave a single saturated furan ester, indicating that such mixtures were simply geometric isomers. It must be pointed out that nuclear magnetic resonance analyses of mixtures of *cis*- and *trans*-isomers were inconclusive, and difficulty in the isomerization of the *cis*- to the *trans*-isomer has been experienced.

Compound V was barely separable on FFAP, but was well separated on SILAR 10C stationary phase. No separation of this mixture was feasible on Apiezon

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\* For Part XVII, see ref. 1.

TABLE I  
EQUIVALENT CHAIN LENGTHS OF SATURATED AND UNSATURATED FURAN ESTERS

Number of carbon atoms in parent chain	Compound	Stationary phase						
		No.	Apiezon L	SE-30	FFAP	DEGS	SILAR 10C	
C <sub>16</sub>		I	16.93	17.00	19.80	21.19	21.90	
C <sub>18</sub>		II	18.94	18.97	21.83	23.08	23.84	
C <sub>16</sub>		III	17.43 (17.81 <sup>**</sup> )	17.36 (17.68 <sup>**</sup> )	21.04 (21.61 <sup>*</sup> )	22.77 (23.46 <sup>*</sup> )	23.86 (24.67 <sup>*</sup> )	
C <sub>18</sub>		IV	19.43 (19.80 <sup>**</sup> )	19.35 (19.65 <sup>**</sup> )	23.11 (23.65 <sup>*</sup> )	24.68 (25.32 <sup>*</sup> )	25.79 (26.54 <sup>*</sup> )	
C <sub>18</sub>		V	18.43	-	21.39 (22.45 <sup>*</sup> )	22.73	23.72 (25.03 <sup>*</sup> )	
C <sub>18</sub>		VI	19.96	19.77	22.80	24.00	24.78	
C <sub>18</sub>		VII	18.98	18.94	22.24	23.67	24.73	

<sup>\*</sup> Baseline separation.

<sup>\*\*</sup> Shoulder separation. ECLs of minor components are given in parentheses.

L and DEGS. The pure sample of compounds VI and VII showed significantly large ECLs.

It can be concluded that the presence of an additional ethylenic bond (conjugated to the furan system or ester function) increases the ECL significantly on both polar and non-polar phases, with the *cis*-isomer contributing a lower fractional chain length (FCL) than that of the corresponding *trans*-compound, the saturated furan ester being regarded as the parent compound.

#### ACKNOWLEDGEMENT

The authors thank the Committee on Higher Degrees and Research Grants of the University of Hong Kong for financial support.

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