# Liquid Crystalline Properties of Cholesteryl $\omega$ -Arylalkanoates

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The thermal properties of the homologous series of cholestery  $\omega$ -(4-benzoylphenyl)- (I),  $\omega$ -(4-benzylphenyl)- (II),  $\omega$ -benzoyl-(III), and  $\omega$ -phenoxyalkanoate (IV) have been investigated. For series I and II the cholesteric-isotropic (Ch-I) transition temperatures, enthalpies, and entropies show a remarkable alternation. For series III and IV, the transition temperatures, enthalpies, and entropies exhibit weak alternation and their trends are opposite to those for series I and II, and the cholesteryl  $\omega$ -phenylalkanoates. The cholesteric-isotropic transition temperatures are discussed in terms of the geometrical and electrical alternations stemming from the terminal aryl groups, and also the relative importance between these two terms.

#### Introduction

A common feature of the even-odd effect on mesomorphic phenomena is that a nematic (cholesteric)-isotropic transition temperature for a mesogen exhibits an even-odd alternation with increasing carbon atoms, n, in an alkyl chain.<sup>1</sup> The transition enthalpy and entropy<sup>2</sup> and also the order parameter<sup>3</sup> have been known to exhibit a similar alternation. These phenomena have been accounted for in terms of the alternation of molecular polarizability in connection with attractive dispersion forces<sup>4</sup> and change in molecular volume.<sup>5</sup> With a bulky substituent such as an aryl group introduced as a terminal function in the alkyl chain, the even-odd effect has been known to become remarkable.<sup>6</sup> For a homologous series having an  $\omega$ -phenylalkyl group, the geometrical alternations of the terminal aryl ring and also the polarity and polarizability have been considered to be of primary importance in determining the transition temperature.<sup>6</sup> In practice, it is not so easy to evaluate the relative importance of these two effects, because these two terms tend to vary simultaneously. The work reported here is intended to provide some information concerning the relative importance of two factors that have been thought to contribute to the remarkable even-odd effect, i.e., the geometrical and electrical alternations stemming from the terminal aryl group. For this purpose, we prepared the following homologous series of cholesteryl  $\omega$ -arylalkanoates.



Series I and II have benzophenone and diphenylmethane groups at the terminal positions, respectively. The characteristics of these series are that the electrical natures, i.e., the dipolarities and

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polarizabilities of the aryl portions, are slightly different from each other, whereas they have similar geometries. Series III and IV have benzoyl and phenoxy groups as the terminal groups, respectively. Series III and IV have geometries similar to those of the cholesteryl  $\omega$ -phenylalkanoates (V), though the electrical properties of the terminal portions are considerably different.

#### Experimental Section

Method. Transition temperatures were determined by using a Nikon Model POH polarizing microscope fitted with a Mettler FP 52 heating stage and FP 5 control unit. Transition enthalpies were measured with a Daini-Seikosha SSC 560 differential scanning calorimeter. Indium (99.9%) was used as a calibration standard with a heating rate of 5 °C/min. Transition entropies were calculated by the equation  $\Delta S = \Delta H/T$ .

Preparation. The method utilized was the same in all cases. As examples, the preparations of the  $\omega$ -(4-benzoylphenyl),  $\omega$ -(4-benzylphenyl),  $\omega$ -benzoyl, and  $\omega$ -phenoxypropionates are given.

 $\omega$ -(4-Benzoylphenyl)propionic Acid. To a solution of benzoyl chloride (0.14 mol) and aluminum trichloride (0.22 mol) in dichloromethane (220 mL) was added methyl  $\omega$ -phenylpropionate (0.14 mol) in dichloromethane (50 mL) at room temperature, and the solution was heated under reflux for 8 h. The resulting solution was poured onto 6 M hydrochloric acid, and the organic layer was collected and washed with water. After the solvent was removed, the residual pale brown solid was heated under reflux in 6 M sodium hydroxide solution for 5 h. The solid obtained by acidification was submitted to column chromatography using silica gel. The column was developed with a mixed solvent of ether and hexane (90:10). The eluent was recrystallized from ethanol, yielding  $\omega$ -(4-benzoylphenyl)propionic acid (12.1 g, 34%) as white needles, mp 94.2 °C.

 $\omega$ -(4-Benzylphenyl)propionic Acid. A mixture of  $\omega$ -(4benzoylphenyl)propionic acid (0.04 mol), potassium hydroxide (0.12 mol), and 90% hydrazine hydrate (9 mL) in diethylene glycol (90 mL) was heated at 110 °C for 2 h. The temperature was gradually raised to 200 °C, the volatile matter being distilled off in the process, and held at this temperature for 8 h. After being cooled, the mixture was poured onto cold water (500 mL) and acidified with concentrated hydrochloric acid. The precipitates were collected and recrystallized from a mixed solvent of ether and hexane, giving  $\omega$ -(4-benzylphenyl)propionic acid as colorless needles (7.2 g, 75%), mp 66.3 °C.

w-Benzoylpropionic Acid. To a solution of succinic anhydride (0.1 mol) in dry benzene (200 mL) was added aluminum trichloride (0.1 mol) at room temperature and the resulting solution was heated under reflux for 5 h. The reaction mixture was poured onto cold 6 M hydrochloric acid. The precipitates collected by filtration were recrystallized from benzene, yielding  $\omega$ -benzoylpropionic acid as colorless needles (9.4 g, 58%), mp 118.2 °C.

 $\omega$ -Phenoxypropionic Acid. A mixture of methyl  $\omega$ -bromopropionate (0.12 mol) purchased from Tokyo Kasei Co, phenol (0.15 mol), and anhydrous potassium carbonate (0.30 mol) in cyclohexanone (150 mL) was refluxed with stirring for 6 h. The

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TABLE I:	Transition	Temperatures,	Enthalpies,	and Entrop	pies of	Series I	and II'

n	Y	T <sub>mp</sub> , °C	$\Delta H_{mp}$ , kJ mol <sup>-1</sup>	$\begin{array}{c} \Delta S_{\rm mp},\\ {\rm J}\ {\rm K}^{-1}\ {\rm mol}^{-1} \end{array}$	T <sub>Ch-l</sub> , °C	$\frac{\Delta H_{\rm Ch-I}}{\rm kJ \ mol^{-1}}$	$\Delta S_{Ch-I}$ , J K <sup>-1</sup> mol <sup>-1</sup>
0	C <sub>6</sub> H <sub>5</sub> CO	134.2	32.9	82.3	210.3	0.50	1.09
1	C <sub>6</sub> H <sub>5</sub> CO	121.1	34.2	86.5			
2	C <sub>6</sub> H <sub>5</sub> CO	93.6	30.4	82.8	117.8	0.67	1.72
3	C <sub>6</sub> H <sub>5</sub> CO				[-35]		
4	C <sub>6</sub> H <sub>5</sub> CO	88.1	30.9	85.7	99.4	1.34	3.59
5	C <sub>6</sub> H <sub>5</sub> CO	66.3	23.1	68.1	(26.2	0.25	0.79)
6	C,H,CO	61.6	22.0	65.6	84.2	1.08	3.01
7	C <sub>6</sub> H <sub>5</sub> CO	35.6	13.6	43.9	40.9	0.33	1.05
8	C <sub>6</sub> H <sub>5</sub> CO	88.0	36.1	99.9	(79.0	0.79	2.26)
9	C <sub>6</sub> H <sub>5</sub> CO	33.9	19.1	62.3	53.4	0.33	1.05
0	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	146.7	36.8	82.3	[100]		
3	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	93.6	40.2	109.7	[-11]		
4	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub>	114.2	36.5	94.2	(59.6)		

"The values in parentheses and square brackets relate to monotopic and virtual transitions, respectively.

TABLE II:	Transition	Temperatures,	Enthalpies,	and Entrop	pies fo	r Series	III and IV	ı

n	x	<i>Т</i> <sub>тр</sub> , °С	∆H <sub>mp</sub> , kJ mol⁻¹	$\Delta S_{mp},$ J K <sup>-1</sup> mol <sup>-1</sup>	T <sub>Ch−I</sub> , °C	$\Delta H_{\text{Ch-I}},$ kJ mol <sup>-1</sup>	$\begin{array}{c} \Delta S_{\text{Ch-I}},\\ J \text{ K}^{-1} \text{ mol}^{-1} \end{array}$
1	СО	127.9	28.0	69.8	(97.7	0.29	0.77)
3	СО	127.4	34.9	87.0	(72.5	0.36	1.05)
4	СО	135.7	38.1	93.2	[105]		,
5	CO	98.7	35.9	98.9	(82.0	0.56	1.58)
6	СО	95.2	36.4	98.7	(98.0	1.52	4.09)
7	СО	75.5	32.1	83.7	(77.3	1.16	3.27)
1	0	122.2	31.4	79.3	(101.0)		·
2	0	110.5	26.1	68.1	[60]		
3	0	107.2	32.9	87.0	(75.3	0.58	1.65)
4	0	108.6	31.0	81.2	(88.1)		•
5	0	84.9	35.4	98.9	(61.5	0.37	1.01)
6	0	103.7	45.0	121.0	(86.8	1.34	3.70)

<sup>a</sup> The values in parentheses and square brackets relate to monotropic and virtual transitions, respectively.

resulting solution was washed with water. After the solvent was removed, the mixture was fractionated by distillation under high vacuum, to give methyl  $\omega$ -phenoxypropionate. The ester was boiled with a mixture of 6 M sodium hydroxide and ethanol (6:4). After acidification, the precipitates were collected by filtration and recrystallized from a mixed solvent of ether and hexane, yielding  $\omega$ -phenoxypropionic acid as colorless needles (10.0 g, 47%), mp 100.5 °C.

Cholesteryl Esters (I-IV). Esterification was carried out by the same method as Gray.<sup>8</sup> Purification was carried out by column chromatography using silica gel. The column was developed with a mixed solvent of ether and hexane (2:98-10:90). Recrystallization was carried out from benzene or a mixed solvent of benzene and ethanol. The purity of the products was checked by thin-layer chromatography using silica gel and elementary analysis.

#### Results

Transition temperatures, enthalpies, and entropies for the homologous series I-IV are summarized in Tables I and II. The cholesteric phases show a typical focal conic texture. Crystallization of the butanoate of series I did not succeed, and the DSC thermogram did not show any peaks in the range between 100 and -50 °C. The butanoate of series I may form a glassy phase. For the pentanoate of II and the butanoate of IV the cholesteric-isotropic transition temperatures could not be detected by DSC because crystallization occurred before the formation of the monotropic cholesteric phase, though the phases could be confirmed microscopically. The values in square brackets in the tables are virtual cholesteric-isotropic transition temperatures, which were determined by an extrapolation procedure using data obtained from binary phase diagrams.<sup>9</sup>



Figure 1. Plot of the cholesteric-isotropic transition temperatures against the number of carbon atoms in the alkyl chain, n, for series I ( $\Delta$ ), II ( $\blacktriangle$ ), and IV ( $\bigcirc$ ).

In Figures 1 and 2 the cholesteric-isotropic transition temperatures for series I-IV are plotted against n and compared with those for the cholesteryl  $\omega$ -phenylalkanoates (V)<sup>66</sup> and alkanoates (IV).<sup>10</sup> There is rather remarkable alternation of the Ch-I

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Figure 2. Plot of the cholesteric-isotropic transition temperatures against the number of carbon atoms in the alkyl chain, n, for series III ( $\phi$ ), V ( $\bullet$ ), and VI (O).

transition temperatures with increasing length of the varied nalkylene group for series I, as well as those for series V. The transition temperatures for the even members of series I are very similar to those for the even members of series V and also of series VI, the only difference being that the transition temperature for the member with n = 0 is relatively higher than that for series V. On the other hand, the transition temperatures for the odd members are slightly lower than those for the odd members of series V. For series II the transition temperatures are likely to show an alternation similar to those for series V, though we have only three data points.

The trends for series III and IV are somewhat complicated. Especially, the transition temperatures for the low members show interesting features. For series IV in Figure 1, the cholestericisotropic transitions give two curves. The one for the even members rises and then tends to fall, and the other for the odd members falls and then rises. The trend in the range between n = 1 and 6 is apparently different from that of the Ch-I transition temperatures for series V. A similar trend is observed in the Ch-I transition temperatures for series III in Figure 2.

The enthalpies and entropies for the Ch–I transitions tend to follow the trend in the transition temperatures. Especially the enthalpies and entropies for homologous series I give rise to marked alternations similar to those for series V.

#### Discussion

It is well-known that the polarizability of a molecule<sup>11,12</sup> and the molecular geometry, i.e., the ratio of the molecular length to the breadth,<sup>13</sup> are the most important factors in determining the thermal stability of a mesophase.

Figure 3 indicates conformational models for the present series where trans rather than cis conformations have been used, because the former results in more linear, rodlike molecular shapes, a general requisite for the mesophase stability.



Figure 3. Conformations of cholesteryl  $\omega$ -arylalkanoates. Dashed lines indicate the cylinder of the steroid portion (diameter ca. 8 Å<sup>15</sup>).

According to Gray's model.<sup>6d</sup> the trend in the even members (model C in Figure 3) is accounted for in terms of the phenyl ring and the substituent of the  $\omega$ -phenylalkyl group lying in the volume of a cylinder defined by rotation of the rest of the molecule about its longer molecular axis (dashed lines). In the odd members (model D in Figure 3), on the other hand, these lie out off the cylinder and increase the molecular breadth, depressing the stability of the cholesteric phase. As is evident from Figures 1 and 2, however, the Ch-I transition temperatures are always lower than those for series VI, except for several low members (n = 0-2). Gray et al. reported that for  $\omega$ -(4-methylphenyl)alkyl and  $\omega$ -(4chlorophenyl) alkyl 4-[(4-cyanobenzylidene)amino]cinnamates the N-I transition temperatures exhibited remarkable even-odd alternation, because the substituents at the phenyl ring were bulker than hydrogen.<sup>6e</sup> Similarly, one can expect that for series I the Ch-I transition temperatures would show more remarkable even-odd alternation than those for series V. As shown in Figure 1, this looks reasonable. However, the Ch-I transition temperature for the pentonate of series II is lower by 40 °C than that for series I, and the butanoate of series II is higher by 24 °C than that for series I. As a result, the difference in the Ch-I transition temperature between the pentanoate and butanoate is only 70 °C for series II, but 134 °C for series I, whereas the terminal groups of two series have very similar geometries. Furthermore, the average Ch-I transition temperature for the octanoate and nonanoate of series V is 66.5 °C and for series I 66.2 °C, whereas the geometrical and electrical circumstances relative to these terminal groups are very different from each other. These facts indicate very clearly that the electrical properties of the aryl groups are also very important in determining the Ch-I transition temperatures.

From the structural point of view, as mentioned above, the diphenyl portion in model A in Figure 3 seems to be more favorable for the stability of the cholesteric phase than that in model B. In addition, it would be reasonable to assume that in model A with Y = -CO- in Figure 3, the terminal benzoyl group enhances the Ch-I transition temperatures, because the dipole of the benzoyl group is directed along the long molecular axis,14 increasing the anisotropy of the dipolarity of the entire molecule. Conversely, the benzoyl group in model B decreases the Ch-I transition temperatures. Accordingly, for the even members of series I, both dipolarity and polarizability, and the preferable geometry of the benzophenone entity, are able to give a greater stability to the cholesteric phase; for the odd members, the circumstances are rather the converse, resulting in a remarkable alternation of the transition temperatures. On the other hand, the benzyl group in model A would tend to decrease the Ch-I

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transition temperatures, because of its relatively small dipolarity which with the polarizability of the benzyl group is directed in a lateral direction in the molecule.<sup>6d</sup> Conversely, the benzyl group in model B tends to increase the Ch-I transition temperatures in spite of the unfavorable geometry. The large difference in the electrical properties between the benzophenone and diphenylmethane entities reflects well in the Ch-I transition temperatures for series I and II.

As is evident from Figure 3, for the even members of series III-V the conformations of the terminal benzoyl, phenoxy, and benzyl groups (model C) are similar to those for the odd members of series I and II (model B). Therefore, the polarity and polarizability of the terminal aryl group for the even members will decrease the Ch–I transition temperatures when X = CO and O (series III and IV) and increase them when  $X = CH_2$  (series V), though their geometrical circumstances are quite similar. Conversely, the electrical circumstances of the terminal aryl groups for the odd members of series III-V (model D) are similar to those for the even members of series I and II (model A).

In model D, therefore, the polarity and polarizability of the aryl group are assumed to increase the Ch-I transition temperatures when X = CO and O and decrease them when  $X = CH_2$  though the geometrical circumstances are quite similar. As shown in Figures 1 and 2, the Ch-I transition temperatures for the low

members of series III and IV lack the characteristic even-odd alternation due to the large contribution of electrical effects by the benzoyl and phenoxy groups.

In conclusion, the electrical and geometrical alternation effects of terminal aryl groups are responsible for the remarkable evenodd alternation of the Ch-I transition temperatures for the low members of series I, II, and V, and the abnormal trends for series III and IV; the former term seems to weaken rapidly with increasing n. The geometrical alternation is then responsible for the even-odd alternation of the Ch-I transition temperatures when *n* is larger, but the effect is gradually averaged when the flexible chain becomes long.

**Registry No.** I (n = 0), 74997-33-4; I (n = 1), 89709-54-6; I (n = 2), 89709-55-7; I (n = 3), 89709-56-8; I (n = 4), 89709-57-9; I (n = 5), 89709-58-0; I (n = 6), 89709-59-1; I (n = 7), 89709-60-4; I (n = 8), 89709-61-5; I (n = 9), 89709-62-6; II (n = 0), 33998-26-4; II (n = 3), 14978-35-9; II (n = 4), 33998-28-6; III (n = 1), 89709-63-7; III (n = 1)3), 89725-93-9; III (n = 4), 89725-94-0; III (n = 5), 89725-95-1; III (n = 5), 89725-1; I = 6), 89709-64-8; III (n = 7), 89725-96-2; IV (n = 1), 89709-65-9; IV (n = 2), 89709-66-0; IV (n = 3), 89709-67-1; IV (n = 4), 89709-68-2; IV (n = 5), 89709-69-3; IV (n = 6), 89709-70-6;  $\omega$ -(4-benzoylphenyl)propionic acid, 71388-83-5; ω-(4-benzylphenyl)propionic acid, 89709-71-7; ω-benzoylpropionic acid, 2051-95-8; ω-phenoxypropionic acid, 7170-38-9.

# **Organic Counterions and Micellar Parameters:** Substituent Effects in a Series of Benzoates

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Critical micelle concentrations obtained by the surface tension method are reported for decyltrimethylammonium micelles in 0.5000 m solutions of 20 aromatic counterions as sodium salts. For meta- and para-substituted benzoates, the principal factor in counterion effectiveness in promoting micelle formation is hydrophobicity. Steric and electronic effects are indiscernible. The effects of ortho substituents involve additional factors which are less clear.

#### Introduction

The mitigation of head group repulsions by bound counterions is an important aspect of micelle formation by ionic surfactants.<sup>1-3</sup> Thus it is strange, in view of the enormous interest in micelles extending over many years, that few studies have involved organic counterions, at least in relation to the large number that might have been examined. Furthermore, the results of such studies,<sup>4</sup>

interesting as each may be, are fragmented by the variety of micellizing amphiphilic ions and the varying goals of the several investigators. Accordingly, we have undertaken a systematic examination of counterion effects based upon decyltrimethylammonium (DTA<sup>+</sup>) as a standard surfactant ion. The decyl chain is long enough for genuinine micelle formation while fewer anions form precipitates with DTA<sup>+</sup> than would be the case with longer chains. The effect of chain length in a series of n-alkyl carboxylate counterions has been reported.<sup>11</sup> In this paper, we discuss critical micelle concentrations (cmc values) for a series of substituted benzoates. As a direct measure of  $\Delta G^{\circ}$  for micellization,<sup>12</sup> the

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