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Liquid Crystalline Properties of 3,4-Di- and 2,4-Di(4-alkoxybenzoyloxy)-benzylidene-4-alkoxyanilines

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This paper describes liquid crystalline properties for some homologs of 3,4-di-(**1**) and 2,4-di(4-alkoxybenzoyloxy)benzylidene-4-alkoxyanilines (**2**) and some related compounds. Compounds **1** tend to show a nematic phase with a notable cybotactic with a layer structure and also a smectic C phase when the alkoxyl groups are suitably long. Compounds **2** show only a monotropic nematic phase, and the nematic–isotropic transition temperatures show a notable even–odd effect even in the higher homologs. The smectic C phase was further characterized by X-ray examination, and the liquid crystalline properties are discussed in terms of the molecular structure characteristic.

Keywords: Synthesis; thermal properties; lateral substituent; X-ray examination; cybotactic; molecular structure

INTRODUCTION

Generally, molecules exhibiting liquid crystalline properties have either a calamitic structure or a discotic one. However, recent works have demonstrated that some molecules having the boundary structure of the two classes show liquid crystalline properties and exhibit interesting physical properties

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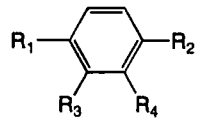
[1–4]. The 1,2,4-tri-substituted benzenes shown below are also one of the classes [5].



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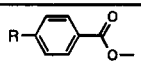
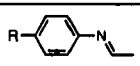
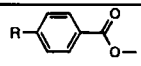
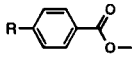
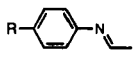
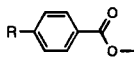
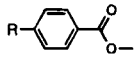
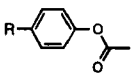
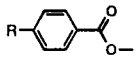
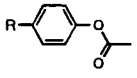
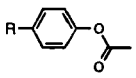
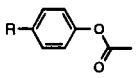
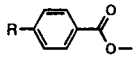
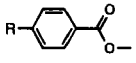
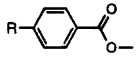
1				H
2			H	
3				H
4				H
5				H

($R = OC_nH_{2n+1}$)

In our earlier paper [5], we described that compounds 3 and 4 involving three ester groups, show liquid crystalline properties despite a wide molecular breadth. 3 show a nematic (N) phase with a shear-like texture, though the nematic-isotropic (N–I) transition temperatures are fairly low compared with those for 4-alkoxyphenyl 4-(4-alkoxybenzoyloxy)benzoate [6]. Thereby, N–I temperatures show a notable even-odd effect. The smectic properties are poor occurring only in the nonyloxy homolog or larger. 4 form the N phase only in the higher members and the N–I transition temperatures are much lower than those for 3. The smectic phase also formed only in the higher members of 4. In contrast, 5 are non-mesogenic, while these have similar structures to those for 3 and 4. These results indicate that in the related systems the relative orientation of the ester linkages is very important in determining the mesomorphic properties. The alternation of the ester linkages should affect both the polarity and geometry of all molecules, since the ester group has a dipole moment of 1.8 D and a flexiblility due to the σ bond nature around the CO—OR and COO—R bonds. On the other hand, an azomethine group is less polar (1.00 D) than

the ester group and has a rigidity due to its double bond. Therefore, the azomethine compounds such as **1** and **2** are expected to show different liquid crystalline properties compared with the ester compounds, **3**–**5**.

In this paper, we describe the syntheses and thermal properties for some homologs of **1** and **2**, and discuss the relationship between the mesomorphic properties and the molecular structures.

EXPERIMENTAL

Materials

3, 4-Di(4-butoxybenzoyloxy)benzaldehyde

A solution of 3, 4-di-hydroxybenzaldehyde (1.0 g, 7.24 mmol) in triethylamine (2.56 g, 25.3 mmol) was added dropwise to the toluene solution (20 ml) of 4-butoxybenzoyl chloride (3.86, 18.1 mmol) at 0°C, and the reaction mixture stirred for 4 h at 80°C. After removing the solvent in vacuum, the reaction mixture was extracted with ether, and the solute was purified by column chromatography on silica gel. The elute was recrystallized from toluene-ethanol, giving 3, 4-di(4-butoxybenzoyloxy)benzaldehyde as colorless needles, 2.01 g (57%): mp 43°C: IR(KBr, cm^{-1}); 1699 and 1740 (ν_{COO}), and ^{1}HNMR ($CDCl_3$, δ(Hz)); 0.98(6H, *t*, J = 7.4), 1.49(4H, sext, J = 7.3), 1.78(4H, quaint, J = 7.0), 3.99(2H, *t*, J = 6.4), 4.00(2H, *t*, J = 6.4), 6.82–6.88(4H, *m*), 7.57 (1H, *d*, J = 8.1), 7.87(1H, *dd*, J = 8.1, 2.0), 7.91(1H, *d*, J = 2.0), 7.97–8.04 (4H, *m*), 10.01(1H, *s*) ppm.

The purity was checked by thin-layer chromatography on silica gel (E. Merck, Silica gel 60 F_{254}), and HPLC (Shimadzu, C-R4XA) where an ODS column was used.

The other homologs were obtained similarly as colorless needles, where the melting points for pentyloxy, hexyloxy, heptyloxy, octyloxy, nonyloxy, and decyloxy homologs are 48, 51, 53, 57, 66 and 68°C, respectively. All the homologs are non-mesogenic.

2, 4-Di(4-butoxybenzoyloxy)benzaldehyde

A similar treatment of 4-butoxybenzoic acid (3.5 g, 18.0 mmol) and 2, 4-dihydroxybenzaldehyde(1.0 g, 7.2 mmol) gave 2, 4-di(4-butoxybenzoyloxy)-benzaldehyde as a colorless oil, 1.8 g (51%): IR(KBr, cm^{-1}); 1693 and 1738 (ν_{COO}), and ^{1}HNMR($CDCl_3$, δ(Hz)); 1.00(6H, *t*, J = 7.4), 1.52(4H, *t*,

$J = 7.3$), 1.81(4H, $q, J = 7.0$), 4.06(2H, $t, J = 6.4$), 4.07(2H, $t, J = 6.4$), 6.98–7.00(4H, m), 7.26–7.32(2H, m), 8.02(1H, $d, J = 9.2$), 8.10–8.19 (4H, m), 10.21(1H, s) ppm.

The other homologs were obtained similarly as a colorless oil.

N-3, 4-Di(4-butoxybenzoyloxy)benzylidene-4-butoxyaniline (1b)

A solution of, 3, 4-di(4-butoxybenzoyloxy)benzaldehyde(0.5 g, 1.0 mmol) and 4-butoxyaniline(0.15 g, 0.9 mmol), and acetic acid(3 drops) in abs. ethanol(25 ml) was heated for 4 h at 70°C. The precipitates obtained by standing overnight were recrystallized from toluene – abs. ethanol, giving **1b** as pale yellow plates, 0.25 g (44%)as: IR(KBr, cm^{-1}); 1734 and 1741 (ν_{COO}), and ^{1}HNMR($CDCl_3$, δ(Hz)); 0.98(6H, $t, J = 7.3$), 0.99(3H, $t, J = 7.4$), 1.45–1.57(6H, m), 1.73–1.81(6H, m), 3.98(4H, $t, J = 6.6$), 3.99(2H, $t, J = 6.4$), 6.82–6.87(4H, m), 6.92(2H, $d, J = 8.9$), 7.23(2H, $d, J = 8.9$), 7.47(1H, d, $J = 8.6$), 7.82(1H, $dd, J = 8.6, 2.0$), 7.94(1H, $d, J = 2.0$), 7.99–8.04(4H, m), 8.48(1H, s) ppm.

The other homologs of **1** were obtained similarly.

N-2, 4-Di(4-butoxybenzoyloxy)benzylidene-4-butoxyaniline (2b)

A similar treatment of 2,4-di(4-butoxybenzoyloxy)benzaldehyde(1.6 g, 3.3 mmol) and 4-butoxyaniline(0.5 g, 3.0 mmol) gave **2b** as pale yellow plates, 0.4 g(21%): IR(KBr, cm^{-1}); 1618($\nu_{C\equiv N}$) and 1736(ν_{COO}), and ^{1}HNMR($CDCl_3$, δ(Hz)); 0.94–1.02(9H, m), 1.41–1.59(6H, m), 1.70–1.86 (6H, m), 3.94(2H, $t, J = 6.4$), 4.03–4.08(4H, m), 6.86(2H, $d, J = 8.9$), 6.95–7.00(4H, m), 7.12(2H, $d, J = 8.3$), 7.23–7.28(2H, m), 8.10–8.17 (4H, m), 8.27(1H, $d, J = 8.3$), 8.60(1H, s) ppm.

The other homologs of **2** were prepared similarly.

METHOD

IR spectroscopy was performed on a Horiba FT-200 infrared spectrometer. ^{1}HNMR spectra were taken with a Nihon-Denshi EX-270 spectrometer in $CDCl_3$, with TMS as an internal standard. Transition temperatures and latent heats were determined, using a Seiko SSC-5200 DSC where indium (99.9%) was used as a calibration standard (mp 156.6°C, 28.4 J/g). The DSC thermogram was operated at a heating or a cooling rate of 5°C/min. Mesophases were characterized using a Nikon POH polarizing microscope

fitted with a Mettler thermo-control system FP-900. A homogeneous alignment between two glass plates was achieved by treatment of the glass surfaces with a polyimide (Tore SP-810), while a homeotropic alignment was achieved by treatment with *N*-trimethyl-cetyl-ammonium bromide.

X-ray diffraction experiments were performed with a Rigaku-denki RINT 2200 diffractometer with CuKα (λ = 1.542 Å) as an X-ray source. The reflection angle (2θ) was calibrated by examination of both left and right, and the temperature of the sample was controlled by a Rigaku PTC-20A thermo-controller. The powdered sample crammed into a quartz capillary (1.5 mm ϕ) was heated up to the isotropic solution, and the measurement was carried out during the cooling process.

RESULTS AND DISCUSSION

Transition temperatures and latent heats for the homologous series of **1** and **2** are summarized in Table I.

The homologs of **1** show a nematic (N) phase with a shlieren or a shear-like texture. **1g** and **1h** also show a smectic phase with a broken focal conic texture under a homogeneous alignment and a schlieren one under a homeotropic one. The latent heats for the smectic-N transition are fairly small. The smectic phase was assigned to the C modification (S_C) from microscopic observation and X-ray analysis.

TABLE I Transition temperatures and latent heats for compounds **1** and **2**

	Transition temperatures (T/°C)								*Latent heats (kJ/mol)*		
	n	*C*		S_C		*N*		*I*	ΔH_{mp}	ΔH_{SC-N}	ΔH_{N-I}
1b	4	•	109	–	(	•	93)	•	41.0		0.5
1c	5	•	89	–	(	•	79)	•	41.2		0.3
1d	6	•	79	–		•	85	•	39.6		0.3
1e	7	•	72	–		•	82	•	52.0		0.2
1f	8	•	65	–		•	84	•	60.9		0.4
1g	9	•	63(	•	43)	•	84	•	51.1	1.8	0.7
1h	10	•	61(	•	56)	•	86	•	63.7	1.6	0.9
2a	3	•	143	–		–		•	50.3		50.3
2b	4	•	91(	–		•	42)	•	47.6		47.6
2c	5	•	80(	–		•	31)	•	47.2		47.2
2d	6	•	65(	–		•	50)	•	47.7		47.7
2e	7	•	71(	–		•	37)	•	45.2		45.2
2f	8	•	83(	–		•	49)	•	76.0		76.0
2g	9	•	88(	–		•	43)	•	82.4		82.4
2h	10	•	90(	–		•	52)	•	77.7		77.7

C, S_C, N and I indicate crystal, smectic C, nematic, and isotropic phases, respectively. Parentheses indicate a monotropic transition.

2 also form a monotropic N phase, and do not show any smectic phase even on rapid cooling. The transition temperatures are plotted against the carbon number of the alkoxy chains in Figure 1.

The phase behavior of the N–I transition temperatures for 1 resembles that of the ester compounds 3 [5] and tend to meet in 85°C, where the even-odd effect is less apparent than 3. On the other hand, the N–I transition temperatures for 2 are fairly low compared with those for 1, and the remarkable even-odd effect is observed throughout the homologs.

The N–I transition temperatures are lower by more than 100°C and the mesomorphisms are very simple compared with those for 4-(4-alkoxybenzoyloxy)benzylidene-4-alkoxyanilines [6], indicating that the 4-alkoxybenzoyloxy group at the 2 or 3 position is very unfavorable for displaying mesomorphic properties. Especially, these large lateral substituents appear to be very unfavorable for smectic mesomorphism.

Usually, a notable even-odd effect in the N–I transition temperature is observed in compounds having two large groups at both terminals of the polymethylene chain, and is interpreted in terms of the packing effect of the terminal groups [7]. The N–I transition temperatures for 2 show a notable even-odd effect, whereas the homologs have no such structural characteristic. We assume that 2 form some peculiar molecular arrangement even in

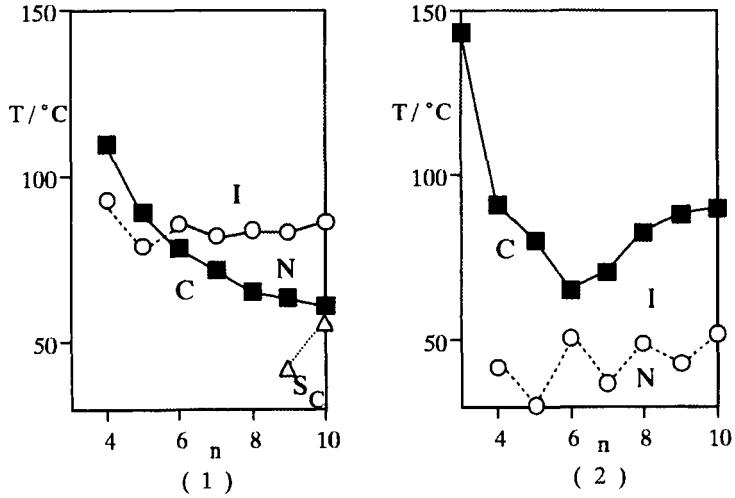


FIGURE 1 Transition temperatures against carbon number (n) for (a) 1 and (b) 2. C, N, S_C, and I indicate crystal, nematic, smectic C, and isotropic phases, respectively. ■: melting point, ○:N–I transition, Δ: S_C–N transition.

the nematic phase, where the terminals of the alkoxyl groups play a very important role in the packing of the molecules.

In order to characterize the mesophases, the X-ray diffraction experiments for **1** were carried out. The X-ray profiles for **1h** are shown in Figure 2.

The X-ray profiles for the N and S_C phases show two reflection peaks. One is a broad reflection peak appearing at 2θ = ca. 20°(3 – 5 Å). This reflection is known to correspond to the average lateral distance between molecules within the N and S_C arrangements (d_{100}). The other appears in ca. 2 – 3°(20 – 40 Å). The reflection peak is observed when molecules form the layer structure (d_{001}) and, usually, the layer spacing of the smectic phase is correlated with the molecular length. The N phase for **1h** shows the reflection peaks in both regions, and the intensity ratio of the peak at the small angle region to the wide one becomes large with declining temperature even in the nematic phase. The maximal position is smaller than that of the peak at the S_C phase, as shown in Figure 2.

The broad reflection at the N phase is observed in every homolog of **1**, and the intensity ratio of the peak at the small angle region (d_{001}) to the wide one (d_{100}) becomes large with an ascending series. These results indicate that an appreciable amount of the cybotactic with the S_C arrangement is present in the N phase.

The X-ray data for **1** and **2** are summarized in Table II.

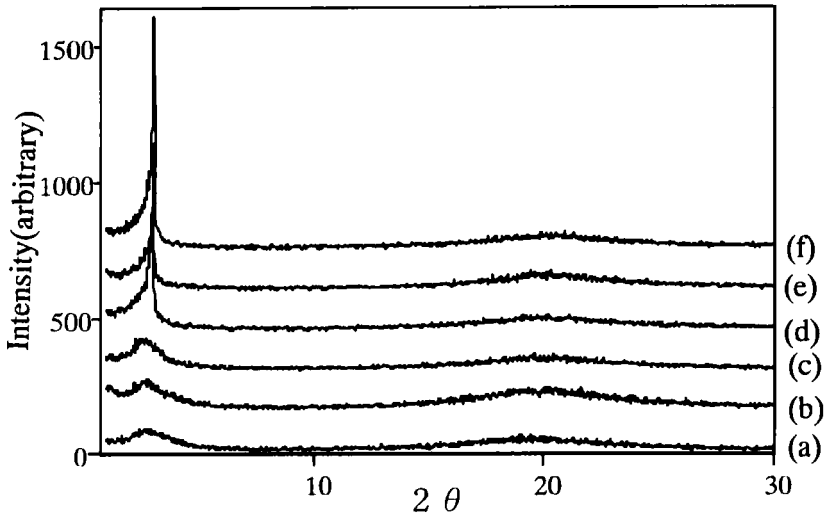


FIGURE 2 X-ray profiles for **1h**. (a) at 80°C (N phase), (b) at 70°C (N), (c) at 60°C (N), (d) at 50°C (S_C phase), and (e) at 45°C (S_C), and (f) at 40°C (S_C phase).

TABLE II Reflection angles and layer spacings for compounds 1, 2, 3, and 4

Compounds	n	Smectic C Phase*1 2θ(degree)	Smectic C Phase*1 d(Å)	Nematic Phase*2 2θ(degree)	Nematic Phase*2 d(Å)
1d	6	–	–	4.0	22
1e	7	–	–	3.5	25
1f	8	–	–	3.3	27
1g	9	3.24	27.2	3.0	29
1h	10	3.04	29.0	2.8	32
2d	6	–	–	4.0	22
2e	7	–	–	3.6	25
3	8	–	–	3.0	29
3	9	3.16	27.9	2.8	32
4	8	–	–	3.0	29
4	9	3.16	27.9	2.8	32
4	10	3.12	28.3	2.7	32

*1 The values were taken at T_{SC-N}-10°C. *2 The values were taken at T_{N-I}-10°C.

The reflection maximum arising from the cybotactic domain increases with an ascending series.

In Figure 2 the reflection peak at the S_C phase becomes sharp with declining temperature, and the maximum changes to 3.02°(29.02 Å) at 50°C, 3.04°(29.0 Å) at 45°C, and 3.06°(28.8 Å) at 40°C. The X-ray data for 1g and 1h are summarized in Table II.

The related ester compounds, 3 and 4, have been reported to exhibit the N and S_C phases [5]. The homologs of 3 and 4 show a similar trend in the X-ray profiles, and the maximal positions are summarized in Table II. As is evident from the table, the layer spacings for the S_C phase of 1 are very similar to those of 3 and 4, indicating that the molecular packing in the N and S_C phases are very similar to each other.

The molecular structures for 1 and 2 were estimated by a semi-empirical molecular orbital calculation (an AM1 method in MOPAC Ver. 6.0). The most stable conformations estimated by the calculation for the decyloxy homologs of 1 and 2 are illustrated in Figure 3.

As we can see from the figure, 1h has a large molecular breadth in the most stable conformation, and it is not possible to approximate either a calamitic structure or a discotic one. In the model, the longest axis passing through the A, B, and C rings is 43.0 Å, which is far longer than the layer spacing (29.0 Å) obtained from X-ray experiments.

It is interesting that such large molecules can be placed in the S_C layer of 29.0 Å without freezing the rotational freedom around the ester, ether, and alkyl groups. A possible model for the packing is that the longest axis of the molecule tilts to the layer, where the large space between the B and D rings involving the terminal alkoxyl groups should be minimized by the closing of

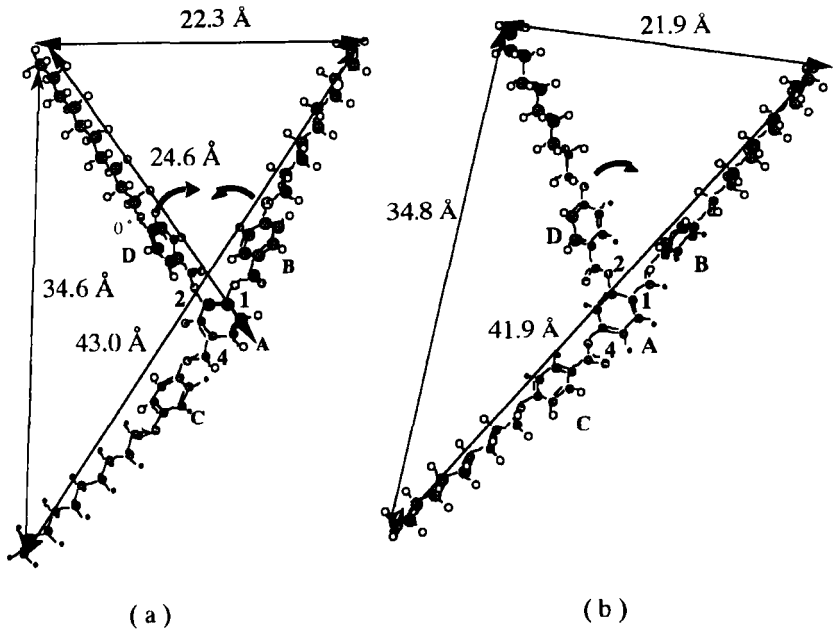


FIGURE 3 Possible molecular structures of (a) **1h** and (b) **2h**, calculated by the AM1 method. The values in the figures are the longitudinal lengths for the most stable conformations.

both moieties, as indicated by arrows in Figure 3(a). The maximal position arising from d_{100} (ca. 20°(4.5 Å)) supports the assumption. In addition, it is also assumed that the dissymmetric molecules form the antiparallel arrangement in order to minimize the molecular volume. The broad and longer reflection peaks observed in the N phase (Fig. 2) might be concerned with the formation of the cybotactic domain with the antiparallel arrangement of the molecules.

2 are assumed to have a similar structure to **1**, as shown in Figure 3(b). Although the homologs of **2** do not show any smectic phase, a similar cybotactic with the layer structure is present in the N phase, as shown in Table II. The higher homologs of **3** and **4** also show very similar X-ray profiles to those of **1** and **2** in both the N and S_C phases, where the reflection angles are almost independent of the difference in the linkages.

These facts indicate that the molecular arrangements in the N and S_C phases are almost independent of the relative orientation of the linkages, though the N–I and S_C–N transition temperatures are strongly affected.

The 1,2,4-trisubstituted benzene core is assumed to be intrinsically unfavorable for maintaining the calaminc structure, whereas, the present compounds and the related ones show liquid crystalline properties, and the

average N–I transition temperatures for the heptyloxy, octyloxy, and nonyloxy homologs of **1**, **2**, **3**, and **4** are 83, 43, 74, and 27°C, respectively, and the homologs of **5** are non-mesogenic, giving the effective order of **1** > **3** > **2** > **4** > **5**. A common fact for **1** and **3** is that 4-alkoxybenzoyloxy groups at the 1 and 2 positions maintain flexibility and rotational freedom around the phenyl —O—CO bonds, and the molecular breadth around these groups can be minimized by conformational distortion without increasing the internal energy, as shown in Figure 3(a). In addition, the ester linkage or the azomethine one at the 4 position increases in the rigidity and linearity along the longitudinal axis passing through the A, B, and C rings, and the calamitic structure of the entire molecule is maintained at the liquid crystalline temperature.

In **2** the ester group involving the D ring locates at the adjacent position of the rigid azomethine bond involving the B ring. The rigidity of the azomethine portion interrupts the proximity of both the B and D rings, as shown in Figure 3(b). Therefore, the molecular breadth of **2** is larger than that of **1**, reducing the N–I transition temperature.

The N–I transition temperature for **4** is lower than those of **1**, **2**, and **3**, while the benzene-1, 2, 4-tricarboxylate core should be the most rigid and plane of all. The rigid phenyl —COO bonds are assumed to destabilize the distorted calamitic structure similar to that in Figure 3(a).

On the other hand, **5** are non-mesogenic, while these have a similar structure to **1**–**4**. **5** have a benzene-1, 2, 4-triol core, where the phenyl —O—CO bonds are too flexible to keep the linearity and rigidity of the longitudinal axis passing through the B, A and C rings.

A similar phenomenon is observed in the 1, 2, 4, 5-tetra-substituted benzenes. Goodby *et al.*, reported that 1, 2, 4, 5-tetra(4-alkoxybenzoyloxy)benzenes show an N phase with uniaxial nature [8], while 1, 2, 4, 5-tetra(4-alkoxyphenoxycarbonyl)benzenes are non-mesogenic [5]. For the former compounds, the flexibility around the phenyl —O—CO bonds might allow the proximity of the adjacent 4-alkoxyphenyl groups as shown in Figure 3(a), and increase the calamitic nature of the entire molecular structure. The core of the latter, benzene-1, 2, 4, 5-tetracarboxylate, essentially have rigidity, similar to **4**, and have a discotic-like shape. The "non-mesogenic" may mean that the benzene-1, 2, 4, 5-tetracarboxylate core is neither a calamitic shape nor a discotic one. Probably, a large steric hindrance between the carboxyl groups distorts the entire molecular structure and reduces the mesomorphic properties, similar to **4**.

In 1, 3, 5-tri-substituted benzene systems, a similar trend is also observed [9]. In our earlier paper, we described that 1, 3, 5-tri(4-alkoxyphenoxycar-

bonyl)benzenes show a monotropic discotic phase, while the bond isomers, 1, 3, 5-tri-(4-alkoxybenzoyloxy)benzenes are non-mesogenic [9]. As we can assume, the core of the former, benzene-1, 3, 5-tricarboxylate, essentially has a discotic shape due to rigidity of the phenyl —COO bond. On the other hand, the core of the latter might have difficulty in maintaining the discotic shape due to flexibility of the phenyl —O—CO bond, similar to **5**.

Conclusively, in 1, 2, 4-tri-substituted benzene systems the molecules must have a considerably hard longitudinal axis in order to display the mesomorphic properties.

Further examination is now underway.

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