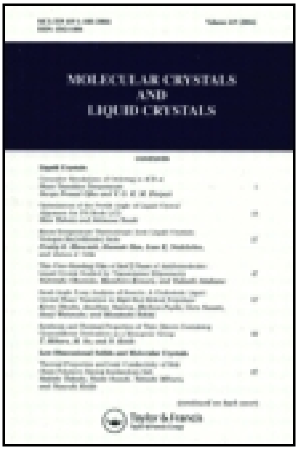

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X-ray Investigation of Smectic Layers in Homologous Series of Chiral Three Ring Esters

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X-ray Investigation of Smectic Layers in Homologous Series of Chiral Three Ring Esters

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Three ring chiral esters, substituted laterally or not with a fluorine atom, having chiral terminal chain of the structure $-COOC^{*}H(CH_3)C_6H_{13}$, *and achiral terminal chain alkylated* $C_nH_{2n+1}COO(CH_2)_mO-$ *or partially fluorinated* $C_nF_{2n+1}COO(CH_2)_mO-$ $(n = 1–7, m = 3–6)$ *were measured by small angle X-ray scattering technique. Most of the fluorinated homologues have high tilted an orthoconic antiferroelectric phase (angle in neighbouring layers is around 90°), which is followed by a SmA phase of like de Vries type. The following parameters are compared: temperature function of d,* d_A, d/l, d_A/l, *(d-layer thickness,* d_A*-layer thickness of SmA phase, l-molecular length) upon temperature and relation with the structure is discussed. The preferred structure for formation of the orthoconic antiferroelectric structure is partially fluorinated terminal alkyl chain and biphenylyl benzoate rigid core. The strongest de Vriesation of SmA phase is observed for longer fluorinated terminal alkyl chain* $(n > 4, m = 6)$ *and phenyl biphenylate rigid core substituted by a fluorine atom in the 2 position of the phenyl ring.*

Keywords: antiferroelectric phase; fluorinated compounds; smectic A de Vries phase; X-ray investigation

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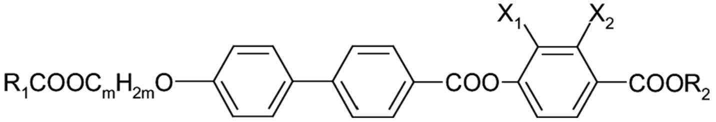
INTRODUCTION

The tilt angle of molecules in the orthoconic antiferroelectric phase is around 45°, thus the conic angle in neighboring layers is around 90°. Such features cause that orthoconic materials are optically uniaxial negative liquid crystals with optic axis perpendicular to the cell glass plates at normal incidence and behave as an isotropic medium at the zero electric fields. Surface defects are not seen which generates the excellent true dark state even when the sample is rotated under crossed polarizers [1–3]. Such high tilt was established by optical measurements, but X-ray measurements of the same materials gave smaller values of apparent tilt angle. It was explained earlier by the formation of SmA phase of de Vries type, and de Vries diffuse model was proposed to explain layer properties [4–8]. In some cases we interpretated such behavior by a process dimerization [9]. The molecules are tilted with respect to the layer normal in the SmA phase, but the tilting direction is randomly distributed there giving uniaxial character of the phase. It may be also consequence so called smectic A block phase (SmA_b) [10]. During the transition to synclinic SmC^* or anticlinic SmC_A^* phases the high tilt angle is produced rapidly by biasing fluctuations and then the tilt angle only increases when temperature decreases. The optical measurements of the tilt in fluorinated esters confirm that the tilt is rapidly generated during the transition SmA-SmC^* [11,12].

Several homologous series of three ring esters with alkyl and fluorinated alkyl terminal chain were investigated by SAXS method to find a relation between the molecular structure and smectic layer structure in orthogonal (SmA) and tilted phases (SmC^* and SmC_A^*). Some such single compounds (mainly from series 1 with spacer $m = 3$) were investigated earlier by us [13,14] and others [15].

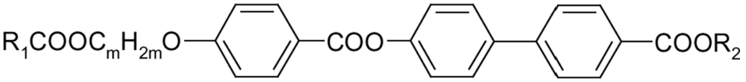
EXPERIMENTAL

Three ring esters of general formula 1 and 2 were investigated:



(1)

series nFmBi ($X_1 = X_2 = H$), nFmBi(2F) ($X_1 = F$, $X_2 = H$) and nFmBi(3F) ($X_1 = H$, $X_2 = F$)


(2)

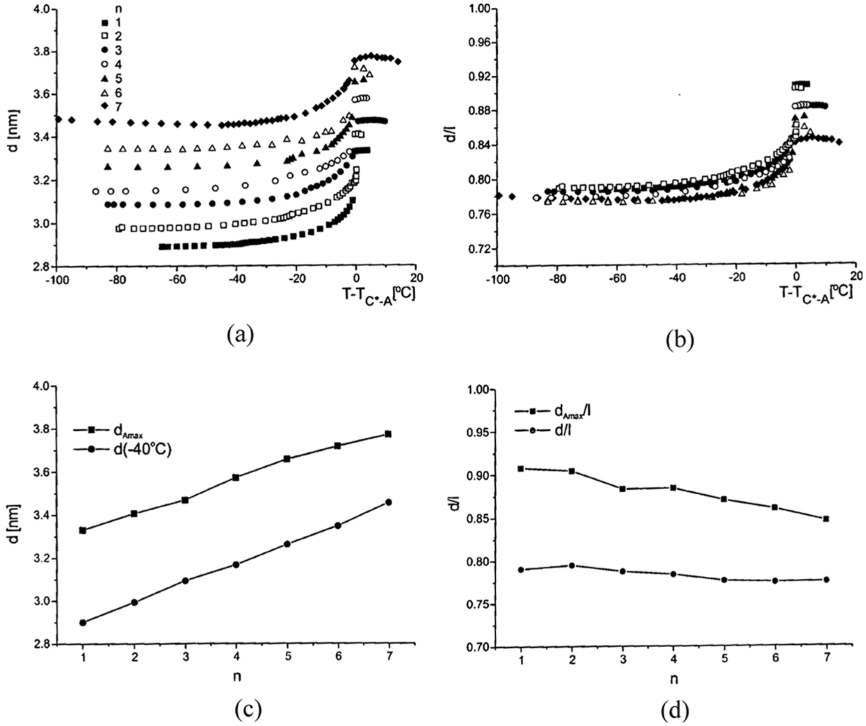
nFmB series

wherein $R_1 = C_nF_{2n+1}$, C_nH_{2n+1}; $R_2 = C^*H(CH_3)C_6H_{13}$, m = 3,4,5,6.

The chain partially fluorinated and non-fluorinated homologous series (1) and (2) are marked respectively nFmBi, nHmBi, nFmB, nFmB. The temperature characteristics of the smectic layer thickness have been studied using the small angle X-ray scattering method (SAXS). An X'Pert (by Philips) powder diffractometer system (with Cu lamp, Ni filter, proportional counter) equipped with hot-stage unit with temperature controller (UNIPAN 660) have been utilized. The results are compared for d_A, d, d_A/l and d/l ratio, where d is layer thickness and d_A is a maximum value of layer thickness in SmA phase, d is given at different temperatures or constant reduced temperature $T - T_{C^*-A} = -40°C$, l is molecular length of all trans molecular conformation calculated by using molecular modeling software HyperChem 5.0 and semi empirical MNDO method.

RESULTS

The dependence of the smectic layer thickness of homologous series nF6B with partially fluorinated terminal alkyl chain upon reduced temperature is shown in Figure 1a as a typical example. The same is presented in Figure 1c, but versus number of carbon atoms n in terminal alkyl chain for a maximum value of layer thickness in SmA phase (d_{Am}) and for layer thickness d in antiferroelectric SmC_A^* phase at fixed $T - T_{C^*-A} = -40°C$ temperature. It is seen that with the increase of alkyl chain length the smectic layer thickness systematically and smoothly increases both in the phase (SmA) as well as in tilted phases (SmC^* and SmC_A^*), and that the smectic layers are shortened rapidly during the transition SmA-SmC^*, and at distance $T - T_{C^*-A} = -40°C$ practically do not depend on temperature. The ratios d/l (reduced layer thickness) in SmA SmC_A^* depend on alkyl chain length (Fig. 1b and 1d) and they are smaller when the compounds have more fluorinated carbon atoms.



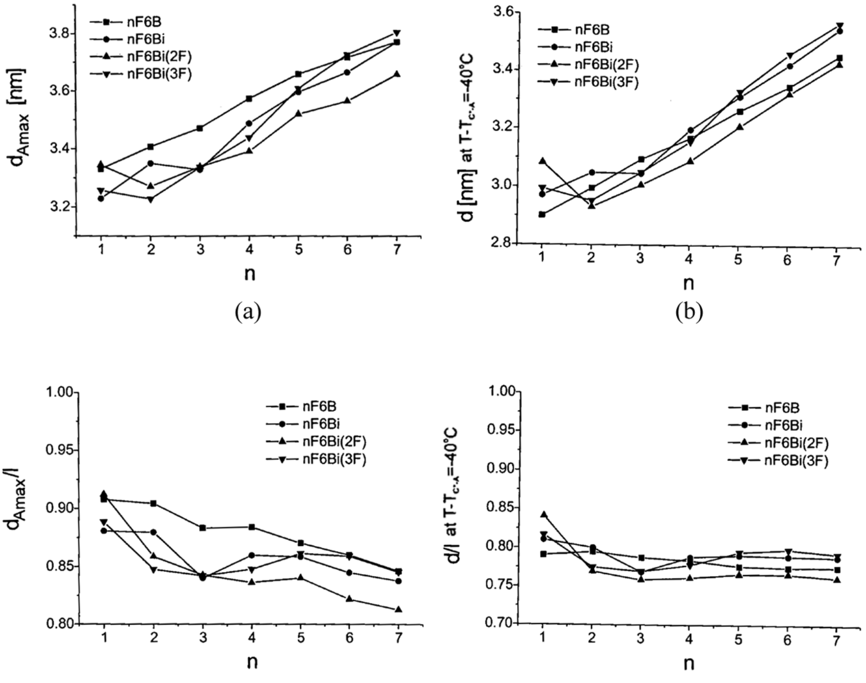
(a) (b)

(c) (d)

FIGURE 1 The layer thickness d (a, c) and the ratio d/l (b, d) upon reduced temperature and versus number of carbon atoms for series nF6B.

The value d/l in smectic A phase for short chain is about 0.9 and fall to about 0.85 for n = 7. Dependence d/l on n is much more significant in the SmA phase; in the SmC$_A^*$ phase at fixed temperature $T - T_{C^*-A} = -40°C$ d/l ratio changes only in 0.8–0.78 range.

The maximum values of layer spacing in smectic A phase (d$_{Am}$) and the ratio d$_{Am}$/l as well as layer spacings d and the ratios d/l in homologous series nF6B, nF6Bi, nF6Bi(2F) and nF6Bi(3F) at fixed $T - T_{C^*-A} = -40°C$ temperature upon length of fluorinated terminal chain unit, presented in Figure 2, let us compare the smectic layer properties for different rigid cores. The layer spacings increase with the elongation of the fluorinated terminal unit both in the SmA and the SmC$_A^*$ phases, Figures 2a and 2b, only for members 1 F6Bi(2F) and 1F6Bi(3F) are some irregularities. The comparison of lateral substitution with a fluorine atom shows that substitution into position 3 in phenyl ring influences on the SmA and SmC$_A^*$ layer spacing only



(a) (b)

(c) (d)

FIGURE 2 The layer thickness in SmA phase d_{Am} (a) and SmC^*_A phase d at $T - T_{C^*-A} = -40°C$ (b) and the ratio d_{Am}/l (c) and d/l (d) versus number of carbon atoms for series nF6B, nF6Bi, nF6Bi(2F) and nF6Bi(3F).

for small n, while substitution into position 2 decreases d_A/l and d/l ratio for the most members especially for members with big n value, see Figures 2c and 2d. In the SmA phase the layer are much shorten for biphenylates series than for benzoates. In the antiferroelectric phase the X-ray layer spacing are less dependent on the structure of rigid core as well the length of fluorinated unit, Figure 2d. Also here members of series nF6Bi(2F) have shorten layers but in the case of benzoate nF6B series the relation is changed and members of this series have a little shorter layer than members of nF6Bi series, except of n = 3, Figure 2d.

The above observation points on the importance of the molecular core interacting.

The influence of fluorination of the terminal chain in biphenyl benzoates and phenyl biphenylates is shown in Figure 3. In both non-fluorinated series nH6B and nH6Bi the smectic layers are much

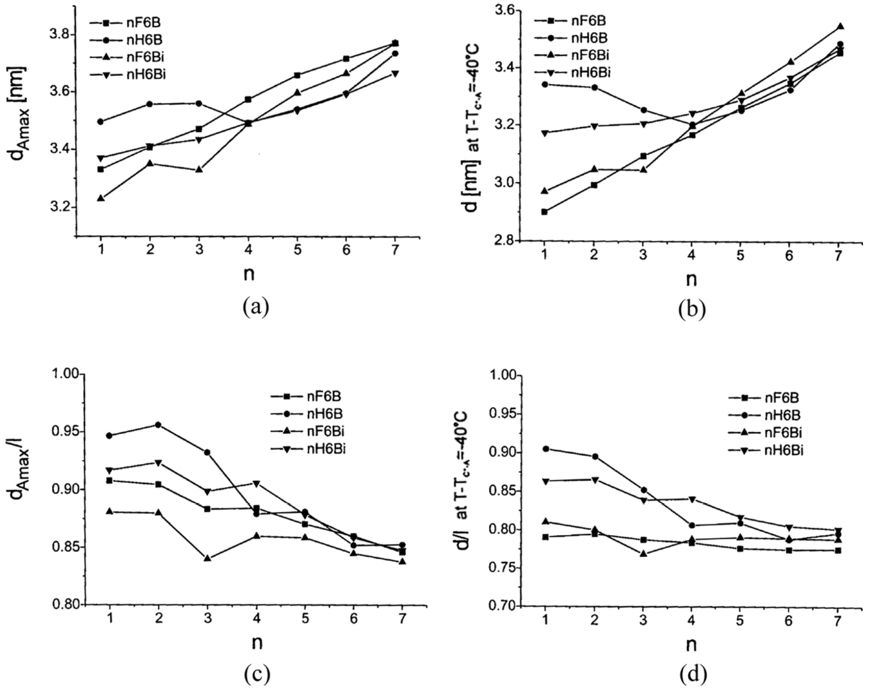
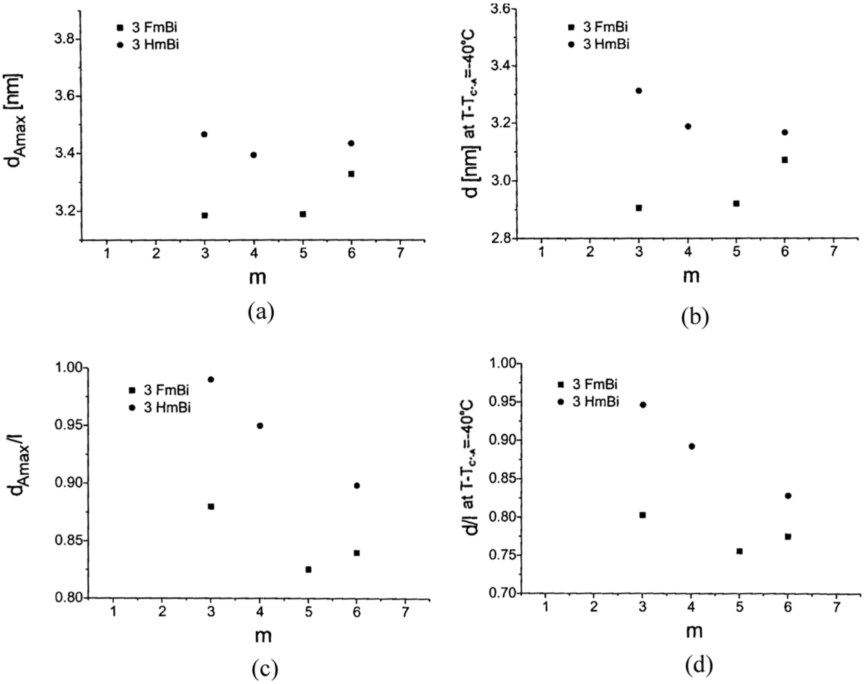


FIGURE 3 The layer thickness in SmA phase d_{Am} (a) and SmC$^*_A$ phase d at $T - T_{C^*-A} = -40°C$ (b) and the ratio d_{Am}/l (c) and d/l (d) versus number of carbon atoms for series nF6B, nH6B, nF6Bi and nH6Bi.

broader for short chains and a little shorter for longer chains both in SmA as well as SmC$^*_A$ phases in comparison to analogous compounds with fluorinated alkyl chains (series nF6Bi and nF6B), Figures 3a and 3b. The ratio d_{Am}/l and d/l in antiferroelctric phase for the non-fluorinated compounds are much bigger than for the fluorinated ones and become more comparable for longer homologues, see Figures 3c and 3d. Figure 3 show that the fundamental differences exist between fluorinated and non-fluorinated compounds in smectic A phase and tilted smectic C* phases for members with short chains and that the differences become smaller for longer chains.

The influence of spacer length on the layer spacing is more significant for compounds with an alkyl chain than for compounds with a fluorinated alkyl chain. The layer spacings decrease with increase n in non-fluorinated compounds Figure 4ab, and therefore the reduced layer spacings (d_{Am}/l and d/l) decrease with the increase of the spacer length from 3 to 6, more than in fluorinated compounds (Fig. 4c and 4d).



(a) (b)

(c) (d)

FIGURE 4 The layer thickness in SmA phase d_{Am} (a) and SmC^*_A phase d at $T - T_{C^*-A} = -40°C$ (b) and the ratio d_{Am}/l (c) and d/l (d) versus number of carbon atoms for series 3FmBi and 3HmBi.

DISCUSSION

The experimental results show, that in the investigated chain fluorinated esters 1 and 2 the smectic layer spacing of the smectic A phase are much shorter than molecular length in the most extended conformation. The ratios d_{Am}/l in the four investigated homologous series is in the rage 0.91–0.81, (see Table 1). So they are smaller than usually observed for the most of monolayer smectic A, having d_{Am}/l between 0.95 and 1 [16]. The layer spacing is only a little differentiated upon rigid core structure, less than inside of the members of the same series. For series nF6Bi, nF6Bi(2F), nF6Bi(3F) and nF6B the ratios d_{Am}/l are changing in the range: 0.88–0.84, 0.91–0.81, 0.89–0.85, 0.91–0.85 respectively.

In fully hydrogenated analogous series nH6Bi and nH6B the ratios d_{Am}/l are a little shifted to a bigger values 0.95–0.85 but meaningful differences between the partially fluorinated and fully hydrogenated

TABLE 1 The Comparison of Smectic Layer Properties in Smectic A (d_{Am}) and Smectic C_A^* Phases (d at $T - T_{C-A} = -40°C$)

Acronym	l	d_{Am}	d	d_{Am}/l	d/l	arccos d_{Am}/l	arccos d/l	d/d_{Am}	arccos d/d_{Am}	θopt Ref. [17]
1F6Bi	3.67	3.23	2.97	0.88	0.81	28.26	35.86	0.92	23.06	
2F6Bi	3.81	3.35	3.05	0.88	0.80	28.38	36.83	0.91	24.53	
3F6Bi	3.96	3.35	3.05	0.85	0.77	32.25	39.75	0.91	24.63	
4F6Bi	4.06	3.49	3.20	0.86	0.79	30.70	37.96	0.92	23.51	
5F6Bi	4.19	3.60	3.31	0.86	0.79	30.84	37.72	0.92	22.87	
6F6Bi	4.34	3.67	3.42	0.84	0.79	32.33	37.86	0.93	20.87	
7F6Bi	4.50	3.77	3.55	0.84	0.79	33.09	37.94	0.94	19.73	
1H6Bi	3.68	3.37	3.16	0.92	0.86	23.52	30.64	0.94	20.22	
2H6Bi	3.69	3.41	3.17	0.92	0.86	22.54	30.94	0.93	21.77	
3H6Bi	3.82	3.43	3.17	0.90	0.83	25.99	33.99	0.92	22.71	
4H6Bi	3.89	3.49	3.21	0.90	0.83	25.96	34.20	0.92	23.09	
5H6Bi	4.02	3.54	3.26	0.88	0.81	28.53	35.94	0.92	22.85	
6H6Bi	4.11	3.59	3.35	0.87	0.81	29.06	35.53	0.93	21.40	
7H6Bi	4.33	3.67	3.45	0.85	0.80	32.03	37.21	0.94	20.04	
1F6Bi(2F)	3.67	3.34	3.08	0.91	0.84	24.18	32.76	0.92	22.80	
2F6Bi(2F)	3.81	3.27	2.93	0.86	0.77	30.79	39.69	0.90	26.38	
3F6Bi(2F)	3.96	3.34	3.01	0.84	0.76	32.57	40.64	0.90	25.79	
4F6Bi(2F)	4.06	3.39	3.09	0.84	0.76	33.26	40.39	0.91	24.38	<45
5F6Bi(2F)	4.19	3.52	3.21	0.84	0.77	32.82	39.98	0.91	24.23	
6F6Bi(2F)	4.34	3.57	3.32	0.82	0.77	34.72	39.97	0.93	21.19	
7F6Bi(2F)	4.50	3.66	3.43	0.81	0.76	35.64	40.35	0.94	20.33	
1F6Bi(3F)	3.67	3.26	2.99	0.89	0.82	27.30	35.25	0.92	23.22	
2F6Bi(3F)	3.81	3.23	2.95	0.85	0.78	32.03	39.18	0.91	23.89	
3F6Bi(3F)	3.96	3.34	3.05	0.84	0.77	32.63	39.66	0.91	23.92	<35
4F6Bi(3F)	4.06	3.44	3.16	0.85	0.78	32.03	38.91	0.92	23.38	
5F6Bi(3F)	4.19	3.61	3.33	0.86	0.80	30.53	37.32	0.92	22.60	
6F6Bi(3F)	4.34	3.73	3.46	0.86	0.80	30.77	37.02	0.93	21.69	

7F6Bi(3F)	4.50	3.81	3.57	0.85	0.79	32.28	37.52	0.94	20.27	
1F6B	3.67	3.33	2.90	0.91	0.79	24.78	37.76	0.87	29.45	35.00
2F6B	3.77	3.41	2.99	0.90	0.80	25.22	37.34	0.88	28.51	43.70
3F6B	3.93	3.47	3.09	0.88	0.79	27.93	38.03	0.89	26.93	37.50
4F6B	4.04	3.57	3.08	0.88	0.76	27.85	40.32	0.86	30.43	44.00
5F6B	4.20	3.66	3.26	0.87	0.78	29.48	39.04	0.89	26.85	43.00
6F6B	4.32	3.72	3.35	0.86	0.78	30.66	39.19	0.90	25.71	42.50
7F6B	4.46	3.77	3.46	0.85	0.78	32.19	39.15	0.92	23.60	43.50
1H6B	3.69	3.50	3.29	0.95	0.89	18.76	26.91	0.94	19.65	
2H6B	3.72	3.56	3.28	0.96	0.88	17.05	28.24	0.92	22.86	
3H6B	3.82	3.56	3.25	0.93	0.85	21.19	31.53	0.91	23.90	
4H6B	3.97	3.49	3.20	0.88	0.81	28.47	36.23	0.92	23.42	
5H6B	4.03	3.54	3.21	0.88	0.80	28.58	37.30	0.91	25.06	
6H6B	4.22	3.60	3.30	0.85	0.78	31.52	38.44	0.92	23.25	
7H6B	4.38	3.74	3.44	0.85	0.78	31.47	38.29	0.92	23.04	

members of the investigated homologous series are only observed for short members ($n = 1 \div 3$). They are almost the same for long members for example $d_{Am}/l = 0.85$ for 7F6B and 7H6B.

Ratios d_{Am}/l smaller than unity may have different reasons. Usually it results that the long molecular axis are not exact parallel each other but they are chaotic tilted a long layer normal (order parameters for smectics A is about 0.9).

Molecules may also diffuse from a layer to a layer. It leads to shorting of layers but for big penetration a dimmer formation may also increase the smectic layer [16]. Molecules may also none exist in the most elongated conformation because the chains may choose different conformation. In the case of chain fluorinated compounds the chain becomes more rigid. The diffusion of molecules between layers and chain interdigitation is hampered. Such situation occurs in the case of the investigated compound as it is confirmed by linear change of the layer spacing upon fluorinated chain length, see Figure 1. Such regularity is not observed for hydrogenated analogues, see Figure 3.

In fluorinated achiral analogues to investigated compounds, but with shorter isopropyl group instead of 1-methylheptyl group in the second terminal position, d_{Am}/l nearly exactly 1.0 [17]. It confirms that interlayer penetration of chains in this case is not observed and molecules exist in the most extended conformation. In these compounds the smetic A phase is strongly ordered as is evidenced by the very big enthalpy of SmA/Iso transition (8–11 kJ/mol) and very high clearing point (>200°C).

Many Authors postulate that for rod-like compounds with only one fluorinated chain, the incompatibility of the hydrocarbon and fluorocarbon chains leads to a segregation of both parts, which may be considered as microphase separation [18–20]. Fluorinated parts of molecules interact between themselves and hydrogenated part between themselves and a lamellar structure or a block structure is formed. For the compounds with one terminally substituted aromatic ring, for example such as methyl heptadecafluorododecan-1-yloxybenzoate the ratio $d/l = 1.35$ because a dimeric antiparallel structure arises [21].

For the compounds with too aromatic rings, monolayer smectic A is favourized, example for trifluoroethyl example heptadecafluorononadecan-1-yloxybiphenylate $d_{Am}/l = 0.77$ [19].

The other reason of shortening of the smectic A layer is such that molecules are more tilted than it results from values of order parameter S, but because they freely rotate the phase behaves as an orthogonal phase. The molecules may also form tilted disorder blocks. Such smectic A phases are known as Adrian de Vries phases [5–7].

1-Methylheptyloxy group is strongly broken against rigid core axis [22], what favourizes higher tilting and hamper the free rotation of molecules in molecular cave especially when the second terminal tails are ordered and strong molecular interactions between themselves and adjacent layers exist.

In fully hydrogenated compounds nH6B with short terminal $C_nH_{2n+1}COO$ group the chains are probably disordered and the observed smectic A phase is a normal A phase. During the transition SmA/SmC* free rotation is space limited and only a small tilt is generated which then slowly increase with the decrease of temperature.

In the case of all fluorinated chain compounds and the hydrogenated ones with higher value n a stronger intermolecular interaction between the chains probably forms microdomain block structure and during the transition SmA/SmC* the blocks are ordered or free rotation of molecules is strongly limited what rapidly generate a big tilt. It is concordant with optical tilt measurements. For members 3F6B, 4F6B and 4FBi(2F) the tilt bigger than 20° degree is generated rapidly during the transition and than it quickly saturate to value 45° degree [13]. Probably for flexible hydrocarbon unit $m = 6$ and more rigid fluorinated unit $n = 3–5$ there are the optimal condition to generate high tilt in molecular cave. Longer fluorinated chain ($n = 6–7$) adopt the optical high tilt slowly and at longer distance from the transition SmA/SmC*. The strong increase of ordering during transition from SmA to SmC phase is confirmed by the increase of birefringence Δn [4].

The best conditions to generate orthoconic tilt do not exist in single compounds but in their mixtures [11,13]. In the mixture of fluorinated compounds is more free space what allows molecules to adopt easily the orthoconic tilt.

Comparing d/l ratios in SmC_A^* (antiferroelectric) phase at the fixed temperature distance 40 degree below the transition SmA/SmC*, when they practically do not depend on temperature (saturated values), we see, Table 1, that they are in the range 0.84 to 0.76, for the all member of fluorinated series. For hydrogenated series they are in the range 0.89–0.78, but for longer members 7H6B and 7F6B are nearly the same. The lowest d/l values 0.76–0.77 are observed for 3F6B and 4F6B members and calculated arcos d/l for this ratio is between 40.5 and 39.7 degree, that is little less than observed optical tilt. It is difficultly explicity to say, if the molecular tilt calculated in such way is real molecular tilt, but it shows a tendency of tilting well correlated with the optical tilt. The optical tilt is not also finally one fixed value, because it depends on the length light used for the measurement [4].

The observation seems to be important, that in the series nH6B and 4F6B for long alkanoyloxy chain (n = 6 and 7) the ratio d/l and d_A/d they are nearly the same in the both series respectively 0.85 and 0.92. High tilt is possible to generate not only in fluorinated chain compounds but also for the compounds (1) and (2) with long hydrocarbon chain. The interaction between many CH_2 groups is probably enough to keep chain in an ordered state and to produce tilted domain structure.

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