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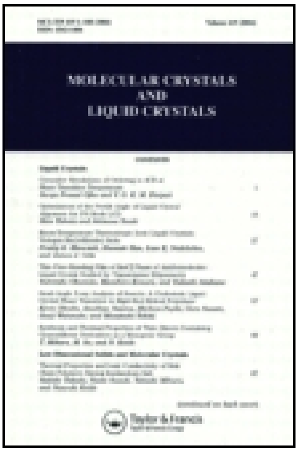

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Effects of Lateral Substituents on Mesomorphic Behavior. IV. Phenyl 4-(4-Alkoxybenzylideneamino)-benzoates

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Effects of Lateral Substituents on Mesomorphic Behavior. IV. Phenyl 4-(4-Alkoxybenzylideneamino)-benzoates

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Phenyl 4-(4-alkoxybenzylideneamino)benzoates exhibit both a nematic phase and a smectic A phase when the alkyl chain is propyl to decyl. While the 2-X substitution tends to eliminate the smectic phase, the 3-X substitution tends to eliminate the nematic phase. A smectic A phase is generated by the methoxy to decyloxy members of the 3-Me, 3-F and 3-Cl derivatives, by the ethoxy and higher members of the 3-Br derivative and by the butoxy and higher members of the 2-F derivative. The smectic thermal stability can be enhanced or depressed by the 3-X substitution depending on the kind of halogen and the alkyl chain length ; for example, 29°C for X=F, 3°C for Cl and – 11°C for Br in the case of the propoxy member and 5°C for X=F, – 15°C for Cl and –24°C for Br in the case of the decyloxy member.

Keywords: lateral substituent; nematic; smectic

INTRODUCTION

The mesomorphic behavior of 4-(4-alkoxybenzylideneamino)phenyl 2- and 3-X-benzoates (1), where X = Me or Cl, were described in our earlier paper [1]. These two lateral substituents are of nearly identical size and lower the nematic-isotropic (N-I) transition temperature by almost the same extent when introduced to the 2-position but differently when introduced to the 3-position, the temperatures for the two substituents being separated by 20°C or more. Furthermore, a smectic A (S_A) phase is generated by the 3-Cl substitution of the higher

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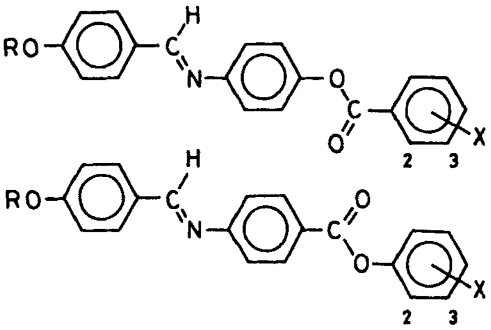
homologous members of the purely nematogenic parent compounds (X = H). We will report here that the behavior of 2-X- and 3-X-phenyl 4-(4-alkoxybenzylideneamino)benzoates (2) differ markedly from that of compound 1 although they are isomeric with each other.

Kuboshita et al. examined the hexyloxy member of the parent compound (X = H) in series 2 and found that the K-S_A, S_A-N, and N-I transitions are located at 103, 143, and 161°C, respectively. The 2-methylation of the phenyl moiety eliminates the S_A phase and the K-N and N-I transitions are found at 95 and 125°C, respectively. On the other hand, the nematic phase is eliminated by the 3-methylation and the K-S_A and S_A-I transition temperatures are reduced to 77 and 117°C [2]. Thus, the mesomorphic behavior of the laterally substituted compounds 1 is seriously modified by the inversion of the carbonyloxy group and a comprehensive work on compounds 2 including the incorporation of halogens into the 2- and 3-positions of the phenyl moiety seemed to be extremely interesting.



1

2

EXPERIMENTAL

All the benzaldehydes and phenols were commercial products. Compound 2 was obtained by condensing 4-aminobenzoic acid with a 4-alkoxybenzaldehyde in refluxing ethanol and then the resulting Schiff's base with a phenol in chloroform at room temperature by the procedure of Hassner and Alexanian [3]. The products were purified by recrystallization from appropriate solvents till sharp constant transition temperatures were recorded. Transition temperature measurements and mesophase identification were performed using a Yanaco melting point apparatus and a Rigaku differential scanning calorimeter as described in a previous paper [1].

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RESULTS AND DISCUSSION

The liquid crystalline transition temperatures and the associated enthalpies of seventy-six compounds are listed in Table I. Here, the crystalline, smectic A, nematic, and isotropic phases are denoted by K, S_A, N, and I, respectively. The N-I transition temperature of each derivative including the parent compounds (X=H) is plotted in Figure 1 against the number of carbon atoms in the alkoxy group. Similar plots of the S_A-N/I transition temperature are presented in Figure 2 where the odd-even alternation is clearly noted. All of the unsubstituted compounds exhibit a nematic phase. Contrary to compound 1 which is purely nematogenic, an additional smectic A phase is observed for the propoxy and higher homologous members; therefore, the present series is suitable to examine the effects of the lateral substituent on the smectic properties.

TABLE I Transition temperatures (°C) and enthalpy changes (kJ mol $^{-1}$) of 2- or 3-X-phenyl 4-(4-alkoxybenzylideneamino)benzoates[a]

n[b]	X	K		S_A		N			I
1	H	.	136 (34)			.	171 (0.3)[d]	[e]	.
2	H	.	163 (46)			.	184 (0.4)[d]	[f]	.
3	H	.	121 (39)	[.	120 (1.3)][c]	.	163 (0.3)[d]		.
4	H	.	115 (32)	.	135 (1.3)	.	168 (0.5)[d]		.
5	H	.	116 (41)	.	135 (1.4)	.	158 (0.4)[d]		.
6	H	.	103 (38)	.	141 (1.6)	.	160 (0.6)[d]	[g]	.
7	H	.	88 (41)	.	144 (2.1)	.	156 (0.6)[d]		.
8	H	.	89 (41)	.	148 (2.5)	.	156 (0.8)[d]		.
10	H	.	92 (48)	.	150[h]	.	151[d]		.
1	2-Me	.	125 (38)			[.	122 (0.3)][c]		.
2	2-Me	.	128 (35)			.	142 (0.4)		.
3	2-Me	.	92 (35)			.	119 (0.3)		.
4	2-Me	.	91 (36)			.	128 (0.4)		.
5	2-Me	.	87 (42)			.	118 (0.4)		.
6	2-Me	.	91 (30)			.	122 (0.5)		.
7	2-Me	.	91 (39)			.	117 (0.6)		.
8	2-Me	.	86 (48)			.	118 (0.7)		.
1	3-Me	.	126 (41)	[.	79 (0.1)][c]	[.	83 (---)][c]		.
2	3-Me	.	117 (36)	[.	104 (2.4)]	[.	113 (0.2)][c]		.
3	3-Me	.	93 (29)	.	96 (3.3)				.
4	3-Me	.	78 (26)	.	114 (3.7)				.
5	3-Me	.	77 (27)	.	109 (3. 2)				.
6	3-Me	.	76 (35)	.	115 (3.4)[j]				.

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n^b	X	K		S_A		N		I
7	3-Me	.	79 (36)	.	114 (3.7)			.
8	3-Me	.	73 (38)	.	119 (4.0)			
1	2-Cl	.	138 (32)			[.	131 (0.4)][c] [k]	.
2	2-Cl	.	146 (39)			[.	142 (0.5)][c]	.
3	2-Cl	.	107 (39)			.	117 (0.3)	.
4	2-Cl	.	96 (38)			.	128 (0.5)	.
5	2-Cl	.	108 (34)			.	117 (0.4)	.
6	2-Cl	.	118 (40)			.	120 (0.5)	.
7	2-Cl	.	114 (39)			.	116 (0.5)	.
8	2-Cl	.	111 (40)			.	117 (0.7)	
1	3-Cl	.	144 (45)	[.	107 (0.6)][c]	[.	112 (0.1)][c] [l]	.
2	3-Cl	.	130 (42)	[.	128 (4.0)[c]	[.	129 (0.3)][c]	.
3	3-Cl	.	97 (36)	.	123 (4.0)			.
4	3-Cl	.	79 (30)	.	133 (4.0)			.
5	3-Cl	.	87 (26)	.	129 (2.9)			.
6	3-Cl	.	93 (38)	.	131 (3.7)			.
7	3-Cl	.	92 (42)	.	132 (4.5)			.
8	3-Cl	.	84 (42)	.	135 (4.8)			.
10	3-Cl	.	87 (47)	.	135 (5.4)			.
1	2-F	.	129 (33)			.	156 (0.4)	.
2	2-F	.	150 (46)			.	168 (0.4)	.
3	2-F	.	113 (38)			.	144 (0.3)	.
4	2-F	.	102 (38)	[.	95 (0.6)][c]	.	150 (0.6)	.
5	2-F	.	101 (30)	[.	90 (0.4)][c]	.	139 (0.6)	.
6	2-F	.	104 (33)	[.	99 (0.7)][c]	.	141 (0.6)	.
7	2-F	.	101 (32)	[.	101 (0.4)][c]	.	135 (0.6)	.
8	2-F	.	90 (34)	.	108 (0.7)	.	136 (0.7)	.
1	3-F	.	119 (34)	.	114 (2.0)	.	154 (0.2)	.
2	3-F	.	137 (38)	.	152 (3.1)	.	165 (0.3)	.
3	3-F	.	106 (24)	.	149 (3.9)			.
4	3-F	.	99 (30)	.	157 (4.7)			.
5	3-F	.	84 (30)	.	152 (4.5)			.
6	3-F	.	108 (42)	.	154 (4.7)			.
7	3-F	.	82 (39)	.	155 (5.3)			.
8	3-F	.	76 (38)	.	156 (5.6)			.
10	3-F	.	82 (46)	.	155 (6.7)			.
1	2-Br	.	133 (34)			[.	105 (0.3)][c]	.
2	2-Br	.	155 (40)			[.	125 (0.3)][c]	.
3	2-Br	.	102 (27)			[.	99 (0.3)][c]	.
4	2-Br	.	95 (31)			.	112 (0.4)	.
5	2-Br	.	105 (36)			[.	102 (0.4)][c]	.

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n^b	X	K		S_A		N		I
6	2-Br	.	116 (40)			[.	108 (0.5)][c]	.
7	2-Br	.	111 (40)			[.	103 (0.5)][c]	.
8	2-Br	.	109 (42)			[.	106 (0.7)][c]	.
1	3-Br	.	144(42)					.
2	3-Br	.	126 (41)	[.	117 (4.2)][c]			.
3	3-Br	.	109 (34)	[.	109 (3.8)][c]			.
4	3-Br	.	91 (31)	.	121 (3.7)			.
5	3-Br	.	82(32)	.	115 (3.4)			.
6	3-Br	.	77 (33)	.	122 (3.6)			.
7	3-Br	.	79 (36)	.	123 (3.9)			.
8	3-Br	.	86(46)	.	125 (4.0)			.
10	3-Br	.	84 (47)	.	126 (4.9)			.

a. The enthalpy changes are in parentheses.
b. The number of carbon atoms in the alkoxy group.
c. Monotropic transition.
d. Taken from Ref. 4.
e. 139 (32) and 174°C (0.4) by Matsunaga and Yasuhara (Ref. 5).
f. 161(36) and 182°C (0.5) by Funakoshi et al. (Ref. 6).
g. 103, 141, and 161°C by Kuboshita et al. (Ref. 2).
h. The combined enthalpy value is 4.6 kJ mol^{-1}.
i. 95 and 125°C and j. 77 and 117°C by Kuboshita et al. (Ref. 2).
k. Taken from Ref. 5.
l. 144 (43) and 113°C (0.2) by Matsunaga and Yasuhara (Ref. 5).

The methyl group incorporated into the 2-position of the phenyl moiety depresses the nematic thermal stability by 49°C when n = 1 and 38°C when n = 8 and removes the smectic phase given by the parent compounds. The same group introduced to the 3-position affects quite differently the mesomorphic properties. First of all, the 3-Me substitution depresses the N-I transition temperature much more than the 2-Me substitution does; for example, 88°C when n = 1 and 71°C when n = 2 (See plots b and c in Figure 1). The opposite is true for compounds 1. Secondly, the higher homologous members are no longer nematogenic. A metastable smectic A phase can be detected even for the methoxy member although the thermal stability of the mesophase of the propoxy and the higher members is reduced by the substitution as much as 21 to 31°C. It may be added that the enthalpies associated with the S-I transition are markedly larger than those associated with the S-N transition of the parent compounds.

The N-I transition temperatures of the 2-Cl derivatives are nearly in agreement with those of the 2-Me derivatives except for the methoxy member for which the 2-Cl derivative gives a temperature higher by 9°C (Compare plots b and f in Figure 1). This situation is similar to what we found for compounds 1. No smectic phase could be detected for the 2-Cl derivatives.

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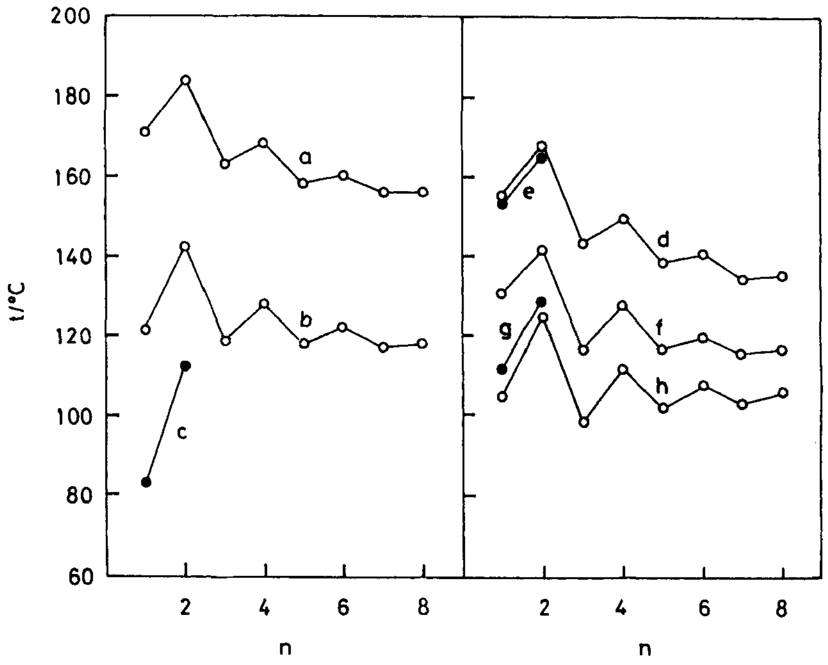

FIGURE 1 Dependence of the N-I transition temperature on the number of carbon atoms (n) in the alkoxy group. (a) the parent compound 2, (b) the 2-Me derivatives, (c) the 3-Me derivatives, (d) the 2-F derivatives, (e) 3-F derivatives, (f) the 2-Cl derivatives, (g) the 3-Cl derivatives, and (h) the 2-Br derivatives

The inversion of the carbonyloxy group is favorable to the generation of the smectic phase in the 3-Cl derivatives. In contrast to compound 1 for which only the hexyloxy and higher members can be smectogenic, all the members of compound 2 exhibit a smectic A phase. Both the S_A-N and N-I transition temperatures of the methoxy member of the 3-Cl derivative are higher by 28 to 29°C than those of the 3-Me derivatives. The difference in the smectic thermal stability decreases to 16°C as the alkyl chain length is increased. The propoxy and higher members are purely smectogenic. Nevertheless, it must be noted that the incorporation of the 3-Cl substituent reduces the smectic thermal stability relative to the parent compounds with exception of the propoxy member for which an enhancement of 3°C is found. The depression of the S-I transition temperature enlarges as the terminal chain length is increased; for example, –2°C for the butoxy member and – 15°C for the decyloxy member (Compare plots a and e in Figure 2).

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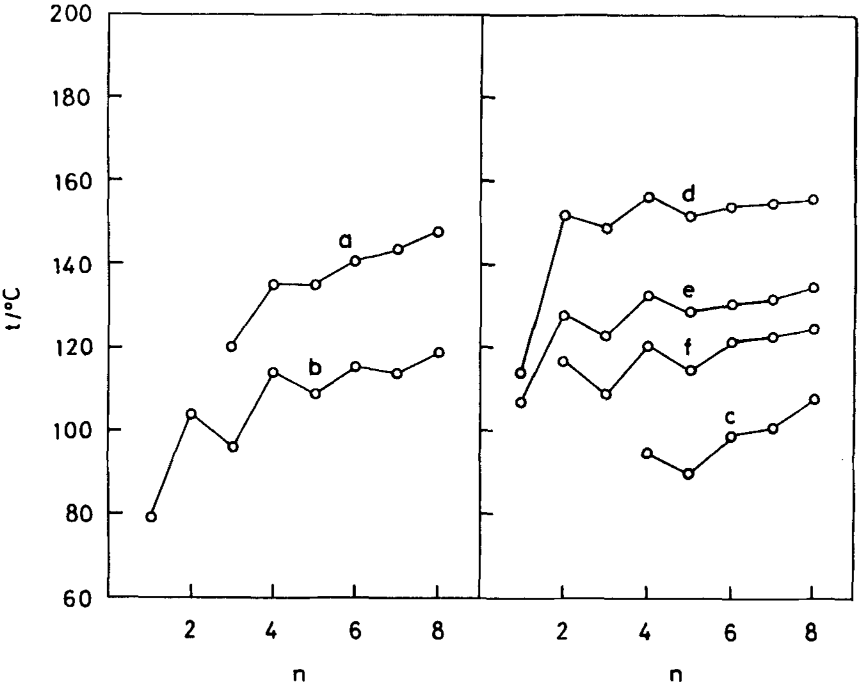

FIGURE 2 Dependence of the S_A-N/I transition temperature on the number of carbon atoms (n) in the alkoxy group. (a) the parent compound 2, (b) the 3-Me derivatives, (c) the 2-F derivatives, (d) the 3-F derivatives, (e) the 3-Cl derivatives, (f) the 3-Br derivatives

The above-mentioned observations differ distinctly from our earlier ones concerning compound 1 that a smectic A phase is generated by the 3-Cl substitution in the purely nematogenic members [1]. In order to back up these conflicting findings, we studied the effects of fluoro and bromo substituents on the mesophase stabilities of the present compounds. The former substituent is expected to enhance more or depress less the smectic thermal stability than the chloro substituent and the latter is expected to depress more.

The 2-F derivatives are nematogenic in conformity with the observation concerning the 2-Cl derivatives. The N-I transition temperatures are lower by 15 to 20°C relative to those of the parent compounds and the depression tends to be larger as the series is ascended (Compare plots a and d in Figure 1). In addition, a smectic phase is observable for the butoxy and higher homologous members even though the thermal stability is lowered as much as 40 to 45°C compared with that of the parent compounds (Compare plots a and c in Figure 2). Accordingly, the S-N transitions are mostly monotropic.

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The N-I transition in the 3-F derivatives is detectable only for the methoxy and ethoxy members, the temperatures of which are lower by a few degrees than those of the 2-F derivatives (See plots d and e in Figure 1). All the members exhibit a thermodynamically stable smectic A phase, the S_A-N/I transition temperature being promoted by 5 to 29°C compared with that of the parent compound (Compare plots a and d in Figure 2). The promotion is the largest for the propoxy member and diminishes monotonously as the alkyl chain is lengthened.

All the members of the 2-Br derivatives exhibit a nematic phase which is generally metastable. The transitions are located at temperatures lower by 50 to 66°C than those of the unsubstituted compounds (See plots a and h in Figure 1). No nematic phase is detected for the 3-Br derivatives. The size of the substituent is so large that the smectic thermal stability is lowered without exception by the 3-Br substitution, making the observation of the mesophase unattainable for the methoxy member. The S_A-N/I transition temperature is more depressed by the 3-Br substitution as the alkyl chain length becomes longer; that is, −11°C for the propoxy member and −24°C for the decyloxy member.

In Figure 3, the N-I transition temperatures of the ethoxy members are plotted against van der Waals volume of the lateral substituents proposed by Bondi [7]. The points for X= H and halogens are on a smooth curve for the 2-X and 3-X derivatives, respectively. The two curves are convex upwards. Contrary to the similar plots for the hexyloxy members of compound 1, the 3-X substituent produces a larger depression than the 2-X substituent. The depression in the N-I transition temperature by the 2-Cl substitution for the ethoxy member is diminished from 82°C for compound 1 to 42°C for compound 2 and that by the 3-Cl substitution is diminished from 60°C for compound 1 to 55°C for compound 2. Thus, the inversion of the carbonyloxy group affects greatly the nematic thermal stability of the 2-Cl derivative but only a little that of the 3-Cl derivative. The point for 2-Me is located distinctly above the curve for the 2-X derivatives but that for 3-Me a little below the curve for the 3-X derivatives.

As to the S_A-N transition temperatures for the octyloxy members of the 3-X derivatives of compound 1, the following values were recorded; 133°C for X=F, 124°C for X=Cl, and 115°C for X=Br [1]. The corresponding values by the present compounds are as follows; 156°C for X=F, 135°C for X=Cl, and 125°C for X=Br. Thus, the smectic thermal stability is markedly enhanced by the inversion of the carbonyloxy group. However, how much the transition temperature is varied by the substitution is a different question. The nematic melt of the octyloxy member of the unsubstituted compound 1 solidified at 90°C without exhibiting any smectic phase. This fact leads to the conclusion that the smectic A phase is stabilized more than 43°C by the 3-F substitution, >34°C by 3-Cl, and > 25°C by 3-Br. On the other hand, the changes in the S-N/I transition temperature

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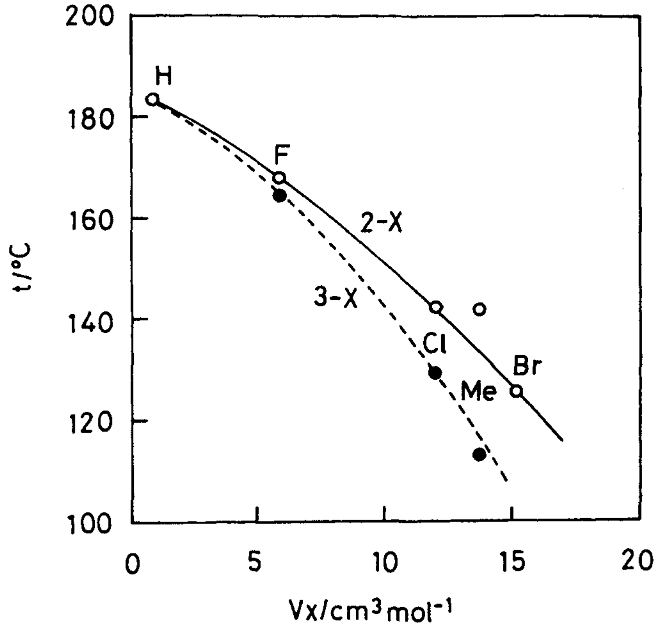

FIGURE 3 Plots of the N-I transition temperature of the ethoxy members against van der Waals volume of lateral substituent

by the 3-X substitution for the same homologous member of compound 2 are 8°C for 3-F, −13°C for 3-Cl, and −23°C for 3-Br.

Lateral substituents may give rise to an increase in the thermal stabilities of a smectic phase by reinforcing the polarity and/or polarizability of the molecule and also a decrease by separating the long axes of the molecules [8]. Of these two opposing effects, the first predominates in the 3-halogeno derivatives of compound 1. As to compound 2, the first predominates only in the 3-F derivatives. The second becomes progressively important as the substituent increases its size as found for most of the 3-Cl derivatives and all the 3-Br derivatives. Thus, the smectic thermal stability is to be enhanced by the 3-Cl substitution when the parent compounds are not smectogenic or the transition temperatures are low but the stability is reduced by the substitution when the transition temperatures are high. This situation resembles closely the one noted by Gray for a range of homologous series [8]. Namely, increasing the alkyl chain length gives a pronounced increase in the S_A-N transition temperature on passing from the octyl to the decyl homologue when the average transition temperature for these homologues is low, whereas the change in the transition temperature is small when the average transition temperature for these homologues is high. He con-

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cluded that when the lateral attractions between the molecules are high giving high S-N transition temperatures, the increased lateral attractions arising from lengthening of the alkyl chain are relatively small in their effect, and vice-versa.

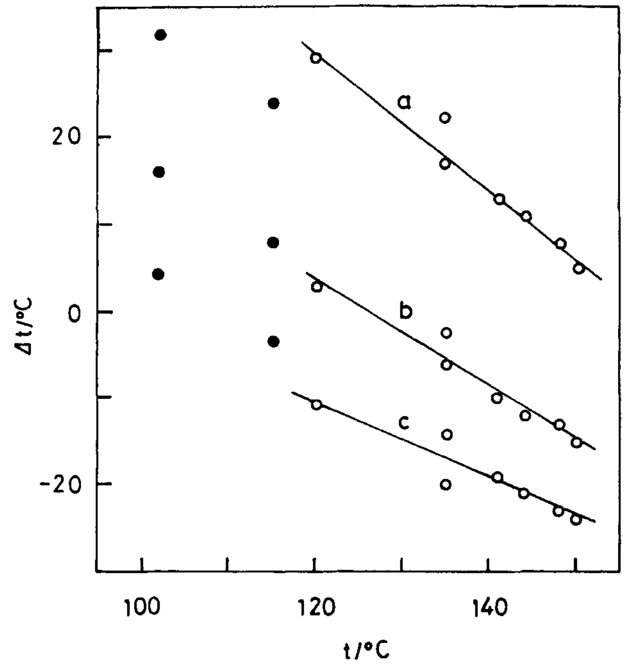

FIGURE 4 Plots of the change in the S_A-N/I transition temperature by the 3-X substitution against the S_A-N transition temperature of the parent compound 2. (a) the 3-F derivatives, (b) the 3-Cl derivatives, and (c) the 3-Br derivatives. The data for the heptyloxy and octyloxy members of *N*-[4- (4-alkoxybenzoyloxy) benzylidene] aniline are indicated by shaded circles

In the present series, the S_A-N/I transition of the parent compound is promoted and the transition enthalpy and entropy become greater as the alkyl chain is lengthened. Therefore, the molecular ordering in the smectic phase is supposed to become higher as the series is ascended. If the molecular ordering in the mesophase of the parent compound is low and/or the size of the 3-halogeno substituent to be introduced is small, the enhancement of the smectic thermal stability by the substitution arising from the increased lateral attractions may be important even if the full size effect of the substituent X operates. When the ordering is high and/or the size of the polar substituent to be introduced is large, the smectic thermal stability may be controlled by the steric disturbance due to the lateral substituent and diminished. Indeed, the change in the transition temperature by the 3-X substitution becomes less positive or more negative as the alkyl chain

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length in the parent compound is increased, in other words, the S_A-N/I transition temperature is raised. The fairly good correlations found in Figure 4, where the changes in the S-N/I transition temperature by the 3-X substitution are plotted against the transition temperature of the parent compound, are in support of our proposition. The straight lines drawn by the method of least squares are a guide for eyes only but seem to approximate well the plots at least in this temperature range. As the S_A-N transition of the parent compound in series 1 is located below 90°C and the associated enthalpy is expected to be small, the increase in the lateral attractions arising from the polar substituent can be as large as estimated above. For comparison, the data for the heptyloxy and octyloxy members of *N*-[4-(4-alkoxybenzoyloxy)benzylidene]aniline, which is isomeric with compounds 1 and 2, are indicated in the same figure by shaded circles [9]. Large deviations from the lines drawn for the 3-F and 3-Br derivatives of the present compounds suggest strongly that the influence of the nature of the molecular core on the correlation cannot be ignored.

4′-Alkoxybiphenyl-4-carboxylic acid studied by Gray et al. is known to be smectogenic when the alkyl group is pentyl or longer, the S_A-N transition temperatures being 230°C or higher [10]. The depressions of the S_A-N transition temperature by various 3′-substituents were discussed by Gray and Worrall employing the data for the octyloxy members [11] ; therefore, it is plausible that the correlation disclosed by them is valid only for well-ordered smectic A phases.

As is demonstrated by the remarkable changes in the mesomorphic behavior by the inversion of the carbonyloxy group of 4-(4-alkoxybenzylideneamino)phenyl benzoates (1), the effects of lateral substituents on mesomorphic behavior are not separable from other molecular parameters determining the intermolecular interaction and the molecular ordering in the mesophase varies from compound to compound. The present work showed how difficult it is to establish the general rule for the effects of a lateral substituent on the mesomorphic behavior, particularly on the smectic behavior.

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