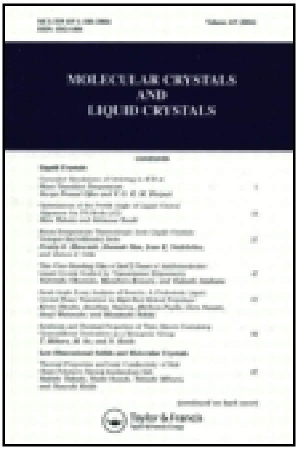

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Chiral Smectic *C Mesogens Having Ester and Amide Central Linkages

R. C. Tandel [a] & R. A. Vora [b]

[a] Chemistry Department, Faculty of Science, M. S. University of Baroda, Vadodara, Gujarat, India

[b] Applied Chemistry Department, Faculty of Technology and Engineering, M. S. University of Baroda, Vadodara, Gujarat, India

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Chiral Smectic *C Mesogens Having Ester and Amide Central Linkages

R. C. Tandel[1] **and R. A. Vora**[2]
[1]Chemistry Department, Faculty of Science, M. S. University of Baroda, Vadodara, Gujarat, India
[2]Applied Chemistry Department, Faculty of Technology and Engineering, M. S. University of Baroda, Vadodara, Gujarat, India

*Ten members of a chiral homologous series 4-(4′-*n*-alkoxy benzoyloxy benzoyl)4″-S- (+) 2-methyl butoxy anilides were synthesized in multiple step synthesis. Series exhibits chiral smectic *C and cholesteric mesophases. In this series, n-propyl to n-dodecyl derivatives exhibit smectic *C and cholesteric mesophases. n-Tetradecyl and n-hexadecyl derivatives exhibit only smectic *C phases. The plot of transition temperatures* versus *number of carbon atoms in alkoxy chain exhibits odd–even effect for cholestric-isotropic transition temperatures. The effect of amide linkage and chirality is discussed in detail. High-temperature cholestric phases exhibit vivid colors. Two compounds were analyzed by calorimetric study (C_{12} and C_{14}) to confirm the transition temperatures of which was observed under polarizing microscope. The mesogenic behavior of the series compared with other series where amide central linkage differs in its position. The mesogenic thermal stability is compared with other related homologous series. The result indicates that amide linkage enhances smectic as well as cholesteric thermal stability but enhancement in smectic phase is much higher. Compounds are characterized by elemental analysis, IR spectral data, Mass spectral data, DSC, NMR, and microscopic study.*

Keywords: chiral smectic *C; ester and amide central linkages; mesogenic; odd–even effect

INTRODUCTION

It is well established that optically active nematogens exhibit cholesteric mesophases. The term “Chiral Nematic” was coined by Dolphin [1] to describe this general class of liquid crystals. Number of chiral nematic liquid crystals have been synthesized based on schiff bases

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Address correspondence to Dr. Ramnik Tandel, Chemistry Department, Faculty of Technology and Engineering, Vadodara, Gujarat 390002, India. E-mail: tandelcy@yahoo.com

[1–4], aromatic esters [5–8], azo-esters [9–11], and biphenyls [12–15]. Chiral nematics, ferroelectric smectic C phases, and chiral smectic A mesogens are of great interest due to their applications in recent times [16,17]. Interest in the synthesis of chiral smectic *C liquid crystals has increased considerably since the advent of a fast switching bistable, electro optical device based on their ferroelectric properties [18–20]. In the early literature, there were few references for mesogenic compounds having amide linkages and terminal chiral linkage. However, to the best of our knowledge , the first homologous series containing (−NH−CO−) amide group as one of the central linkages was reported by Vora and Gupta [21,22]. Soon after that, Kalyvas and McIntyre [23] reported a symmetrical homologous series having an ester and amide central linkages. Subsequently, Brown [24] has also mentioned that compounds with −CONH− may exhibit mesomorphism. It was thought of interest to reverse the amide linkage and to study its effect on the mesomorphic properties of such homologous series and the effect of chirality on a similar no chiral mesogenic series; with this in view it was planned to synthesize chiral mesogenic homologous series with reverse amide linkage compared to earlier studies and explore the possibility of obtaining chiral nematic (cholesteric), chiral smectic *C phases, and classical smectic and nematic phases.

In the last few years, several mesogens and model compounds are reported having amide and ester linkages [25–30].

EXPERIMENTAL

The route adopted for the synthesis of chiral mesogens is shown in Figure 1. The purity of the final compounds was checked by the thin-layer chromatography (TLC) method.

Microanalysis of compounds was recorded on Coleman Instrument. IR spectra and Fourier Transform Infrared Spectrometer (FTIR) spectra were recorded on Perkin–Elmer spectrophotometer and NICOLET Impact-400 FTIR, respectively. NMR spectras were recorded on Hitachi NMR R-1200 Instrument. Liquid crystalline properties were investigated on Leitz-Labourlux 12Pol (Germany) polarizing microscope provided with kofler heating stage. Differential Scanning Calorimetry (DSC) were investigated on Mettler TA 4000 system. Electrospray Ionization Mass Spectrometry (ESI-MS) were recorded on a LC-MS Shimadzo-2010 Instrument.

Procedure

4-*n* Alkoxy benzoic acids and 4-n alkoxy benzoyloxy benzoic acids 9–11 were synthesized by the method described in the literature.

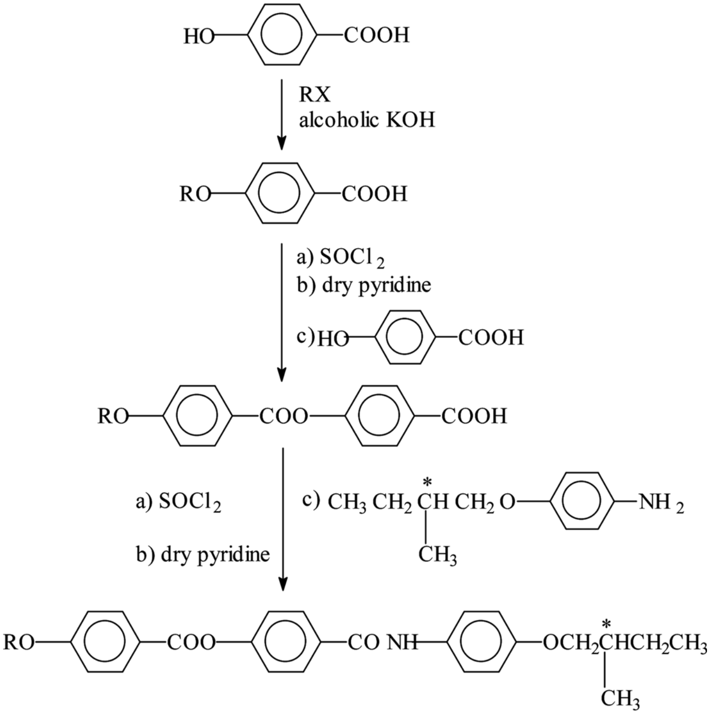


FIGURE 1 Synthetic route to homologous series I. Here, R = *n*-C m H 2m + 1, m = 3 to 8, 10, 12, 14, and 16.

S(+)-1-bromo-2-methyl butane was synthesized from S(+) 2-methyl butanol (12) and S-(+)-4-(β-methyl butoxy) anilines were synthesized by the method described in the literature [31].

Preparation of 4-(4′-*n*-Alkoxy Benzoyloxy Benzoyl)4″-S (+)-β-methyl Butoxy Anilides

Respective 4-*n*-alkoxy benzoyloxy benzoyl chloride (0.01 mole) was dissolved in about 5 ml of dry pyridine. The solution of S-(+)-4 (β-methyl butoxy) aniline (0.01 mole) in 10 ml of dry pyridine was added slowly to cold solution of acid chloride of respective 4-*n* alkoxy benzoyloxy benzoyl chloride. The mixture was heated on water bath for about half an hour and was allowed to stand overnight. It was acidified with cold 1:1 hydrochloric acid. Precipitates were filtered and washed by water followed with sodium hydroxide and water. The resulting ester-amides were recrystallized several times from Dimethyl Formamide (DMF) until constant transition temperatures were obtained

TABLE 1 (4′*n*-Alkoxy benzoyloxy benzoyl)4″-S-(+)-β-methyl butoxy anilides

		Transition temperatures °C		
Sr. No.	R = *n*-alkyl group	Smectic	Cholesteric	Isotropic
1	Propyl	175	273	300 (d)
2	Butyl	202	264	316 (d)
3	Pentyl	214	260	308 (d)
4	Hexyl	218	272	315 (d)
5	Heptyl	214	269	301 (d)
6	Octyl	207	264	306 (d)
7	Decyl	194	236	294
8	Dodecyl	174 (168.3)	193.5 (190.9)	256 (254.7)
9	Tetradecyl	153 (151.1)	–	213 (209.5)
10	Hexadecyl	139	–	193

(d) Decomposition, () indicates DSC data.

(Table 1). The yield of purified ester-amides varies between 65–70%. The synthesized compounds give satisfactory elemental analysis (Table 2).

Spectral Analysis of 4-(4′-*n*- Alkoxy Benzoyloxy Benzoyl)4″-S (+)-β-Methyl Butoxy Anilides

IR(KBr) Spectrum (n-Prpyloxy Derivative)

3325 cm^{-1} and 1645 cm^{-1} –NH stretching and bending of secondary amide, 1600 cm^{-1} stretching vibration of C=O of amide, 1510 cm^{-1} V_{C-H} bending of alkene (–CH_2–), 750 cm^{-1} V_{C-H} bending of aromatic ring, 1730 cm^{-1} carbonyl stretching of aromatic ring of ester, 2900 cm^{-1} and 1415 cm^{-1} V_{C-H} stretching, and bending vibrations of –CH3, 1600 cm^{-1} and 1210 cm^{-1} of aromatic ring. Other signals are observed at 1165, 820 cm^{-1}, etc.

TABLE 2 Average Thermal Stabilities °C

Series	(I) R′ = $^*C_5H_{11}$	(A) R′ = $^*C_5H_{11}$	(B) R′ = C_4H_9
Cholesteric/Nematic to Isotropic (C_1 to C_8)	307.0 (C_3–C_8)	200.17	256.75
Smectic-Chol./or to Nematic/Isotropic (C_6–C_{16})	238.0	199.83 (C_{12}, C_{14}, C_{16})	–
Commencement of Smectic mesophase	C_3	C_{12}	–

IR(KBr) Spectrum (n-Octyloxy Derivative) (Fig. 2)

3325 cm^{-1} and 1644 cm^{-1} –NH stretching and bending of secondary amide, 1600 cm^{-1} stretching vibration of C=O of amide, 1515 cm^{-1} V_{C-H} bending of alkene (–CH2–), 750 cm^{-1} V_{C-H} bending of aromatic ring, 1730 cm^{-1} carbonyl stretching of aromatic ring of ester, 2900 cm^{-1} and 1410 cm^{-1} V_{C-H} stretching, and bending vibrations of –CH3, 1600 cm^{-1} and 1210 cm^{-1} of aromatic ring. Other signals are observed at 1165, 810 cm^{-1}, etc.

FTIR(KBr) Spectrum (n-Decyloxy Derivative) (Fig. 3)

3346 cm^{-1} and 1648 cm^{-1} –NH stretching and bending of secondary amide, 1604 cm^{-1}. Stretching vibration of C=O of amide, 1514 cm^{-1} V_{C-H} bending of alkene (–CH_2–), 759 cm^{-1} V_{C-H} bending of aromatic ring, 1735 cm^{-1} carbonyl stretching of aromatic ring of ester, 2924 cm^{-1} and 1414 cm^{-1}, V_{C-H} stretching, and bending vibrations of –CH3, 1604 cm^{-1} and 1206 cm^{-1} of aromatic ring. Other signals are observed at 1165, 827, 690 cm^{-1}, etc.

ESI-MS were recorded on a LC-MS Shimadzo-2010 instrument and mass values are recorded as:

Mass spectrum (*n*-pentyloxy derivative):

[$M^+ + NH_4^+$, relative intensity (%)]
144.2(24), 160(78), 202(100), 219(41), 247(17), 438(17.5), 495(15).

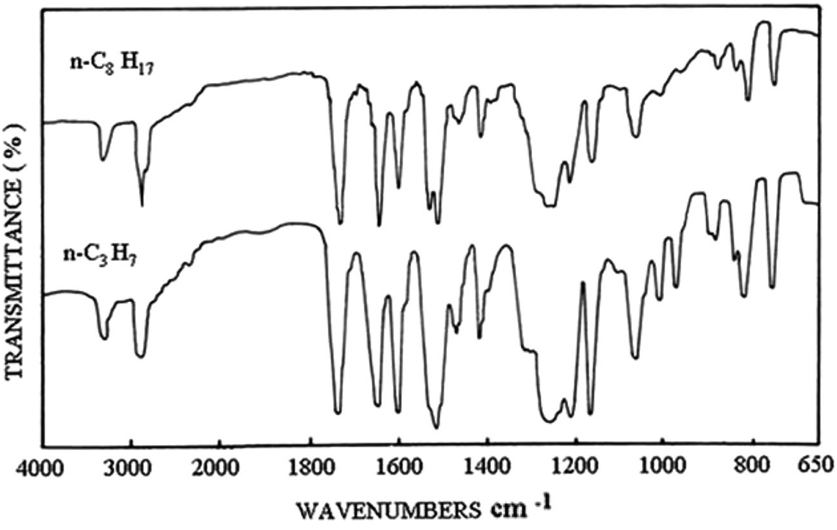


FIGURE 2 Representative IR spectrum of 4-(4′*n*-alkoxy benzoyloxy benzoyl) 4″-S (+) -β-methyl butoxy anilides for compounds R = n-C_3 H_7 and n-C_8 H_{17}.

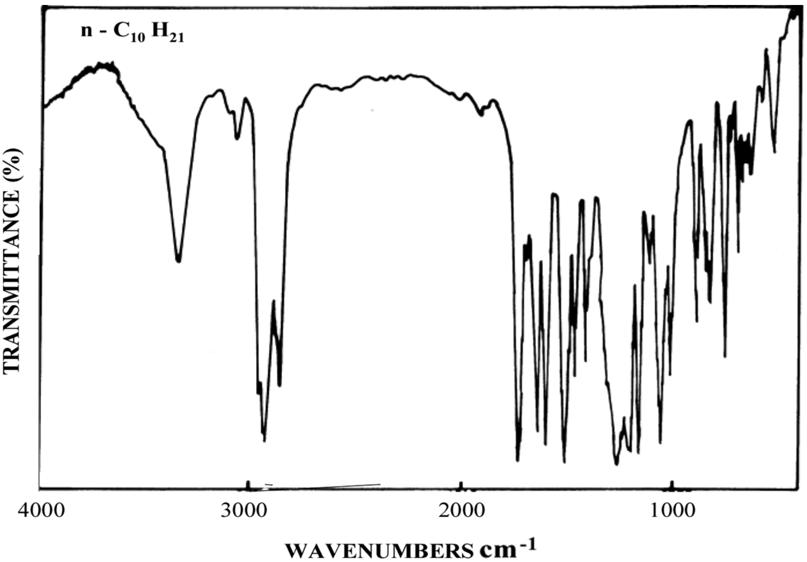


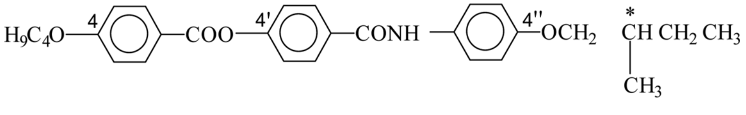
FIGURE 3 Representative FTIR spectrum of 4-(4′-*n*-decyloxy benzoyloxy benzoyl)4″-S (+)-*β* -methyl butoxy anilide.

Mass spectrum (*n*-decyloxy derivative):

[$M^+ + Na^+$, relative intensity (%)]
137(28), 165(80), 208(100), 213(8), 243(4.5), 341(5), 429(6), 560(20), 582(22).

NMR Spectra (n-Butoxy Derivative) (Fig. 4)

^{1}H NMR (90 MHz, $CHCl_3$)



1.0–1.79 (m, 16H of alkyl chain at C-4 and C-4″);
4.2 (t, 4H of two methylene group in the alkoxy chain at C-4 and C-4″)
7.11 (d, 4H at C-3, C-5, C-3″ and C-5″)
7.46 (d, 4H at C-3′, C-5′, C-2″ and C-6″)
8.17 (d, 2H at C-2′ and C-6′)
8.32 (d, 2H at C-2 and C-6)
8.9 (S, 1H of –CO–NH– of amide linkage).

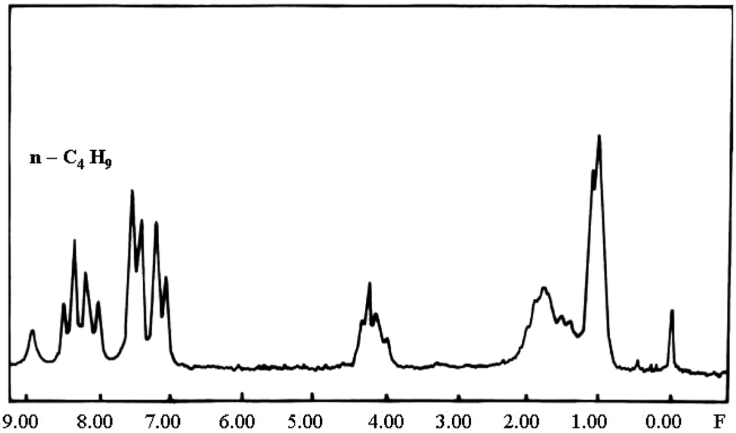


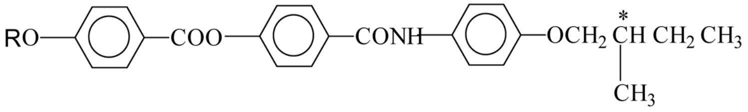
FIGURE 4 ^{1}H NMR spectrum of 4-(4′-*n*-butoxy benzoyloxy benzoyl)4″-S (+)-*β*-methyl butoxy anilides.

RESULTS AND DISCUSSIONS

Vora *et al.* [31,32] and Gupta [33] took up the study of number of mesogenic series with ester and amide central linkages and also of mesogenic series having two amide linkages. The observation of Vora *et al.* made it clear that compared to an ester and azomethine linkage, amide-central linkage enhances smectic and nematic phases, and enhancement is more in case of smectic mesophase.

It has been observed by Griffin and others [16,17] that reversal of the amide central linkage exhibits some remarkable differences in liquid crystalline properties. It was thought proper to synthesize a homologous series having reversal of amide linkage to compare with other similar homologous series reported by our school. Moreover it was thought proper that reversal of the amide linkage may induce smectic C phase in the series which would be highly useful if it is further converted to chiral smectic *C by introducing chiral substituent in the system.

With this in view, the following homologous Series I was synthesized and their mesogenic properties were studied.



SERIES I

Here, $R = (n\text{-}C_m H_{2m+1})$ and m = 3 to 8, 10, 12, 14, 16.

For the synthesis of series I, intermediate 4-*n*-alkoxy benzoic acids and 4-*n*-alkoxy benzoyloxy benzoic acids were synthesized, which are mesogenic in nature. The transition temperatures of all the intermediate acids compare well with the literature value with a few exceptions. The introduction of a chiral tail in the molecule will give chiral smectic *C phase which imparts ferroelectric properties to the molecules.

The series is synthesized as indicated in Figure 1. Homolog from n-propyl derivatives onwards exhibit smectic *C and cholesteric mesophase up to *n*-dodecyl derivative. *n*-Tetradecyl to *n*-hexadecyl derivatives exhibit only smectic *C phases.

The plot of transition temperatures against number of carbon atoms in alkoxy chain for series I exhibits odd–even effect for cholesteric-isotropic transition temperatures as well as for smectic-cholesteric transition temperatures (Fig. 5).

The odd-even effect in the molecules, forming liquid crystals, generally possesses the basic skeleton of relatively rigid aromatic or alicyclic rings with alkoxy or alkyl chains attached to one or both the ends. Within the isotropic or nematic phases, different regions of each molecules are continuously in contact with parts of neighbouring

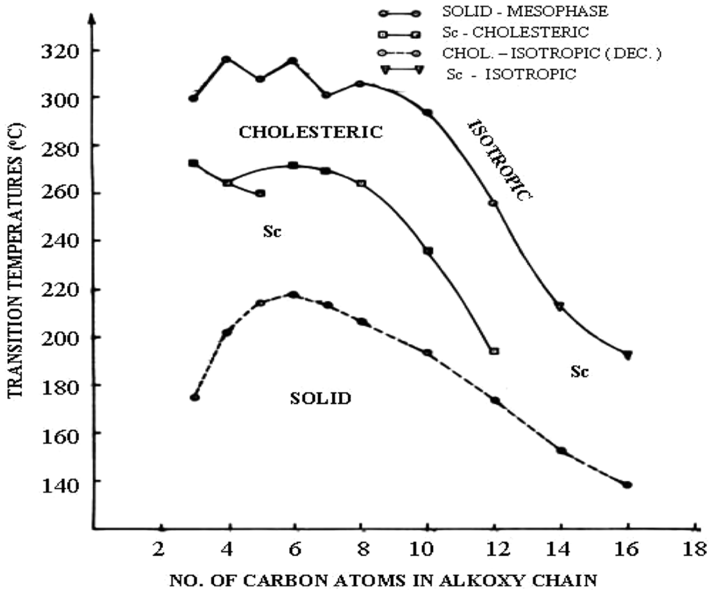


FIGURE 5 Graph of transition temperatures versus number of carbon atoms in alkoxy chain.

molecules. Gray [36,37] has explained the odd-even effect based on the geometry of alkoxy or alkyl chain.

The transition temperature curves fall steeply, whereas solid-mesomorphic transition temperature curve exhibits rising tendency initially, and then falls steeply (Fig. 5).

A typical behavior of transition temperature curves is observed in the graph (Fig. 5). They fall steeply. In the same way, solid-mesomorphic transition temperature curve exhibits rising tendency initially, and subsequently falls steeply. Normally, mesomorphic homologous series do not exhibit such behavior. The odd–even effect is observed for cholestric-isotropic transition temperatures as well as for smectic–cholesteric transition temperatures.

There is a close relationship between mesomorphism and molecular constitution of organic compounds. Hence, thermal stability, a measure of mesomorphism, can be correlated with the molecular constitution of the compounds.

The average thermal stabilities are compared to different homologous series (Table 3). Average thermal stabilities of 4-(4′-*n*-alkoxy benzoyloxy benzoy)4″-S(+)-β-methyl butoxy anilides (series I) are compared with those of.

1) S(+)-4(4′-β-methyl butoxy benzoyloxy)-4″-*n*-alkoxybenzoyloxy benzanilides. [A] [38]
2) 4-(4′-*n*-alkoxybenzoyloxy)-4″ -*n*-butoxy benzanilides. [B] [39]

The reference to the geometry of series I and series A (Fig. 6) indicates they are essentially the same except that one of the central

TABLE 3 Elemental Analysis

		Calculated (%)			Found (%)		
Sr. No.	R *n*-alkyl group	C	H	N	C	H	N
1	Propyl	72.89	6.72	3.04	72.60	6.78	3.13
2	Butyl	73.26	6.95	2.95	72.84	7.10	2.87
3	Pentyl	73.62	7.16	2.86	73.51	7.06	2.70
4	Hexyl	73.96	7.36	2.78	73.65	7.14	2.69
5	Heptyl	74.28	7.54	2.71	74.17	7.46	2.66
6	Octyl	74.58	7.72	2.64	74.38	7.08	2.86
7	Decyl	75.13	8.05	2.51	75.34	8.13	2.61
8	Dodecyl	75.64	8.35	2.39	74.79	7.79	2.58
9	Tetradecyl	76.10	8.62	2.28	75.70	8.36	2.31
10	Hexadecyl	76.52	8.87	2.18	76.35	8.90	2.09

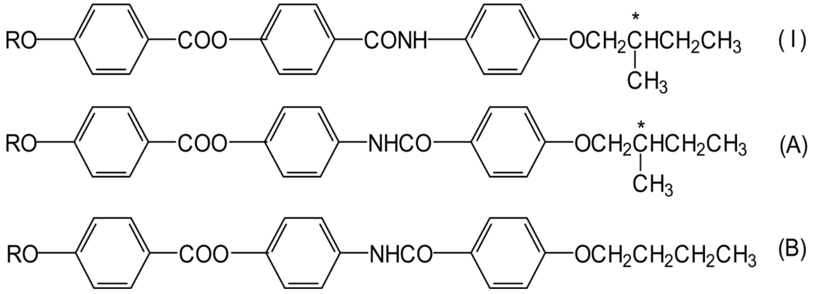


FIGURE 6 The molecular geometry of present series (I) and comparative series (A and B).

linkage amide (–CO–NH–) is reversed in series A as –NH–CO– in the molecules. In the present series, the nitrogen of amide linkage is attached to terminal phenyl ring and can take part into group reasonating structures along with terminal 2-methyl butyl group which contribute to polarizability of the molecules of series I. In the case of series A, the nitrogen of amide linkage is attached to central phenyl group which is attached to another phenyl ring via an ester linkage. This would cause a difference in the polarizability of molecules of series I and A. This is also reflected in the thermal stabilities of both homologous series. The commencement of smectic mesophase is delayed in series A which is at dodecyl derivative. The S(+) β-methyl butyl group at one end does induce smectic tendency in a system, however reversal of amide linkage has made it more pronounced. The molecules of series I and series B differ in reversal of amide linkage and in fixed *n*-alkoxy group at one end which is *n*-butoxy in the case of series B and S (+) β-methyl butoxy group in the case of series I. Series B does not exhibit smectic phase in the homologous series but it commences from the *n*-propyl derivative in series I. Danuta et al. [41] have synthesized a number of model compounds with ester-amide linkage to understand mesogenic behavior of polyesteramides. Their results indicate that reversal of amide linkage in the model compounds where nitrogen of amide linkage is attached with terminal phenyl ring exhibits higher thermal stability.

Two members (C12 and C14 of series I) were screened by DSC method. Both members exhibit prominent endothermic peaks for solid-mesogenic transitions, small peaks for smectic-cholesteric and mesogenic-isotropic transitions. Both members are showing endothermic peaks for phase transitions. Enthalpy and entropy values are recorded in Table 4. Both members are showing endothermic peaks as well as exothermic peaks on heating and cooling, respectively (Figs. 7 and 8).

TABLE 4 4-(4′*n*-Alkoxy benzoyloxy benzoyl)4″-S-(+)-β-methyl butoxy anilides

Calorimetric data

Sr. No.	R = n-alkyl group	Weight (mg)	Transition state	Peak temps. (microscopic reading)	ΔH J/G	ΔS J/g.°K	Total ΔS J/g.°K
1	Dodecyl	12	K–Sc	168.3 (174.0)	11.70	0.0271	
			Sc–Chol	190.9 (193.5)	2.10	0.00463	0.0389
			Chol–Iso	254.7 (256.0)	3.73	0.00721	
2	Tetradecyl	10	K–Sc	151.1 (153.0)	24.06	0.057	
			Sc–Iso	209.5 (213.0)	4.91	0.0177	0.0747

In the present study of the type of smectic mesophase and cholesteric mesophase are inferred from the textures observed under polarizing microscope “Leitz Laborlux Mciroscope” provided with heating stage (Figs. 9 and 10).

CONCLUSION

A new mesogenic homologous series with chiral center have been synthesized. *n*-Propyl to *n*-dodecyl derivatives exhibit smectic *C and

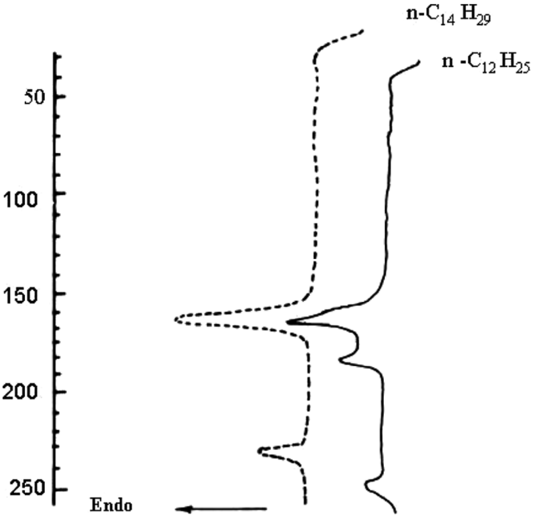


FIGURE 7 Representative DSC thermogram of 4-(4′-n’alkoxy benzoyloxy) 4″-S(+)-2-methyl butoxy anilides for compounds R = *n*-C_{14} H_{29} and *n*-$C_{12}H_{25}$ on heating rate at 10°C min^{-1} in N_2 gas.

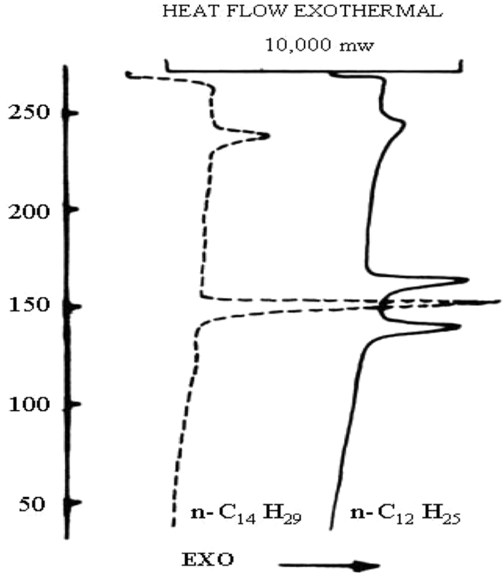


FIGURE 8 Representative DSC thermogram of 4-(4′-*n*’alkoxy benzoyloxy) 4″-S(+)-β-methyl butoxy anilides for compounds R = n-C_{14} H_{29} and n-C_{12} H_{25} on cooling rate at 10°C min^{-1} in N_2 gas.

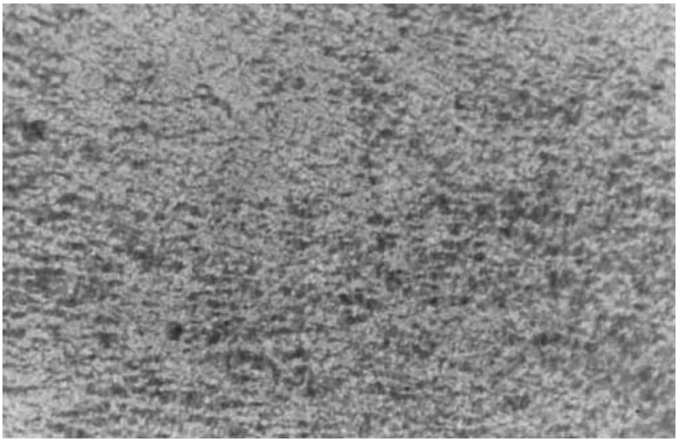


FIGURE 9 Optical polarized microphotograph of cholesteric liquid crystalline phase of 4-(4′-*n*-decyloxy benzoyloxy benzoyl) 4″-S-(+)-β-methyl butoxy anilides at 270°C on heating.

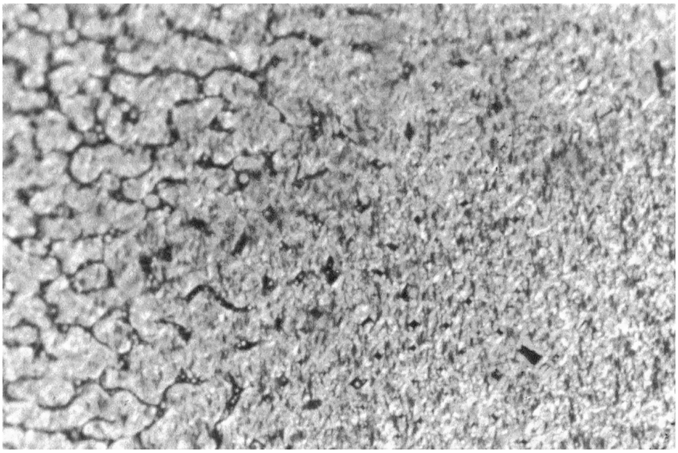


FIGURE 10 Optical polarized microphotograph of smectic and cholesteric liquid crystalline phases of 4 - (4′-*n*-decyloxy benzoyloxy benzoyl) 4″-S-(+)-β-methyl butoxy anilides at 215°C on cooling.

cholesteric mesophases. n-Tetradecyl and *n*-hexadecyl derivatives exhibit only smectic *C phases. The plot of transition temperatures *versus* number of carbon atoms in alkoxy chain exhibits odd–even effect for cholestric-isotropic transition temperatures as well as smectic–cholesteric transition temperatures. Compounds exhibit smectic and cholesteric phases due to chiral center in a molecule as well as ester and amide central linkages.

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