Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713926090

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To cite this Article Vora, R. A. , Prajapati, A. K. , Kevat, J. B. and Raina, K. K.(2001) 'Mesogenic properties and the effect of 1,2,4-trisubstitution on the central benzene nucleus of a three-ring mesogen', Liquid Crystals, 28: 7, 983 — 989

To link to this Article: DOI: 10.1080/02678290110048273

URL: http://dx.doi.org/10.1080/02678290110048273

LIQUID CRYSTALS, 2001, VOL. 28, NO. 7, 983–989

Mesogenic properties and the effect of 1,2,4-trisubstitution on the central benzene nucleus of a three-ring mesogen

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(*Received 21 May 2000; in final form 10 September 2000; accepted 21 September 2000*)

Four mesogenic homologous series have been synthesized by fixing a rigid 4-substituted phenylazo group to a resorcinol moiety. In series I and II, one phenolic $-$OH group is esterified by 4-*n*-alkoxybenzoyl groups; in series III and IV both the phenolic $-$OH groups are esterified by 4-*n*-alkoxybenzoyl groups. All the homologues of series III and IV exhibit low melting smectic C phases. Monoesters of series I and II, having free lateral hydroxy group with strong hydrogen bonding, exhibit high temperature nematic phases. The effect of different substituents on mesomorphic properties is discussed. One homologue of series III was doped with a chiral dopant and its spontaneous polarization evaluated.

1. Introduction

Most thermotropic liquid crystals are rod-like molecules having a rigid core composed of two or more aromatic and/or alicyclic rings and one or more flexible terminal alkyl/alkoxy chains [1]. Laterally substituted mesogens are of considerable interest because these compounds deviate from the classical rod-like shape. After an initial investigation by Weissflog and Demus [2], a number of homologues series with a trisubstituted benzene nucleus have been reported [3–10]. However lateral hydroxy groups are little exploited as it was reported that the phenolic ‘hydroxy’ group may destroy mesomorphism, due to strong intermolecular hydrogen bonding [11]. Schroeder and Schroeder [12] reported a few terminally hydroxy substituted mesogens. Vora and Gupta [13] for the first time reported homologous series with terminal and lateral hydroxy groups. Subsequently a few more mesogenic series with a lateral hydroxy group have been reported [14–16]. In the present study two mesogenic homologous series (I and II) containing a lateral hydroxy group have been synthesized.

Bayle *et al.* [14] reported mesogens with lateral substituents having one azo linkage and diesters with an aromatic nucleus. They observed that when the lateral aromatic branch is on the inner ring of the molecules in one series and on the outer ring in another series, not only did the transition temperatures differ markedly but the type of mesophase also changed. In general, a rigid lateral substituent perturbs the ordering of the liquid crystalline phase [17–19], causing a significant depression in the clearing point and liquid crystal phase transition. However, the effect of a flexible lateral substituent, such as a substituted aromatic moiety, is quite different [2–4]. In order to investigate the influence of the lateral aromatic branch, we have synthesized two mesogenic homologous series (II and IV) containing three rings in the main core and substituted by a lateral aromatic branch on the central ring.

2. Experimental

2.1. *Characterization*

Microanalysis of the compounds was performed on a Coleman carbon–hydrogen analyser, and IR spectra were recorded on a Schimadzu IR-408 instrument. NMR spectra were recorded on a Perkin-Elmer R-32 spectrometer. Liquid crystalline properties were investigated using a Lietz Laborlux 12 POL microscope provided with a heating stage. DSC was performed on a Mettler TA-4000 system.

2.2. *Synthesis*

The synthetic routes to series I–IV compounds and the chiral dopant are illustrated in the scheme.

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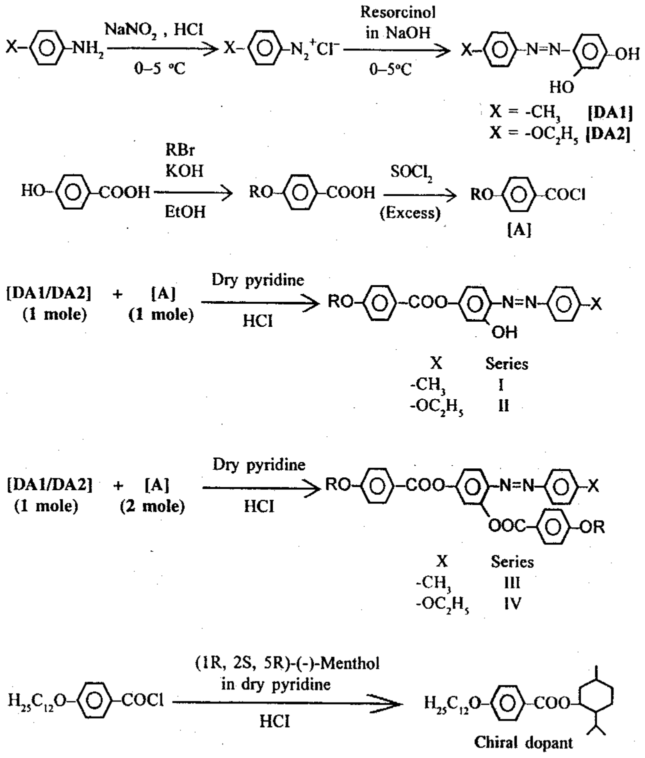
Liquid Crystals ISSN 0267-8292 print/ISSN 1366-5855 online © 2001 Taylor & Francis Ltd
http://www.tandf.co.uk/journals
DOI: 10.1080/02678290110048273



Scheme. Synthetic routes to series I–IV and chiral dopant.

4-Methyl-2′-4′-dihydroxy azobenzene (DA1) and 4-ethoxy 2′,4′-dihydroxy azobenzene (DA2) were synthesized using conventional methods of diazotization and coupling [20]. 4-*n*-Alkoxybenzoic acids and 4-*n*-alkoxybenzoyl chlorides were synthesized by the modified method of Dave and Vora [21]. The twentyfour monoesters of series I and II were prepared by condensing equimolar quantities of 4-*n*-alkoxybenzoyl chlorides with DA1 and DA2, respectively in dry pyridine. The twentyfour diesters of series III and IV were synthesized by condensing two moles of 4-*n*-alkoxybenzoyl chlorides with one mole of DA1 and DA2, respectively in dry pyridine [21]. All the monoesters of series I and II were crystallized from acetic acid; the diesters of series III and IV were crystallized from alcohol until constant transition temperatures were obtained. The transition temperatures of series I and II are recorded in table 1, those of series III and IV are recorded in table 2. The elemental analysis of all the compounds was found to be satisfactory (tables 3 and 4). IR and NMR spectral data are given below.

Table 1. Transition temperatures (°C) for series I and II compounds.

n	N	I
Series I		
1	173.0	254.0
2	134.0	257.0
3	144.0	248.0
4	132.0	248.0
5	120.0	240.0
6	119.0	238.0
7	117.0	225.0
8	118.0	214.0
10	111.0	196.0
12	104.0	182.0
14	104.0	178.0
16	103.0	172.0
Series II		
1	135.0	259.0
2	145.0	252.0
3	140.0	240.0
4	120.0	236.0
5	110.0	225.0
6	104.0	220.0
7	107.0	215.0
8	112.0	210.0
10	106.0	201.0
12	110.0	193.0
14	105.0	187.0
16	105.0	182.0

IR (KBr) spectra for *n*-hexyloxy derivatives of series I and III and *n*-butyloxy derivatives of series II and IV: series I 3600–3200 (−OH), 2900, 1740 (−COO−), 1605 (−N=N−), 1510, 1480, 1415, 1310, 1250, 1180, 1080, 830, 750 cm^{-1}; series II 3600–3200 (−OH), 2900, 1730 (−COO−), 1610 (−N=N−), 1505, 1480, 1410, 1310, 1245, 1180, 1065, 845, 755 cm^{-1}; series III 2950, 1730 (−COO−), 1610 (−N=N−), 1510, 1480, 1420, 1300, 1250, 1170, 1050, 830, 750 cm^{-1}; series IV 2955, 1735 (−COO−), 1605 (−N=N−), 1505, 1480, 1425, 1300, 1250, 1165, 1055, 845, 760 cm^{-1}.

NMR spectra (solvent $CDCl_3$, standard TMS): series I (*n*-butyloxy derivative, 90 MHz) δ 0.95 (t, 3H, $-CH_3$), 1.3–1.9 (m, 4H, $2\times -CH_2-$), 2.3 (s, 3H, $-PhCH_3$), 3.9 (t, 2H, $-OCH_2$), 6.75 (d, $J=9$ Hz, 2H at C-3′ and C-5′), 7.1 (d, $J=9$ Hz, 2H at C-3 and C-5), 7.4–7.8 (m, 5H at C-2, C-6, C-6′, C-3″ and C-5″), 7.95 (d, $J=9$ Hz, 2H at C-2″ and C-6″), 13.4 (s, 1H, −OH); series II (*n*-tetradecyloxy derivative, 300 MHz) δ 0.95 (t, 3H, $-CH_3$), 1.3–1.5 (m, 24H, $12\times -CH_2-$), 1.8 (t, 3H, $-O-C-CH_3$), 4.05 (t, 2H, $-OCH_2-$), 4.1 (q, 2H, $-OCH_2-$), 6.8–7.0 (m, 4H at C-3, C-5, C-5′ and C-6′), 7.1 (s, 1H at C-3′), 7.8 (d, $J=9$ Hz, 2H at C-2 and C-6), 8.0 (d, $J=9$ Hz, 2H at C-3″ and C-5″), 8.15 (d, $J=9$ Hz, 2H at C-2″ and C-6″); series III (*n*-butyloxy derivative, 90 MHz) δ 0.95 (t, 6H, $2\times -CH_3$), 1.4–1.9 (m, 8H for $4\times -CH_2-$), 2.3 (s, 3H, $-PhCH_3$), 4.0 (t, 4H, $2\times -OCH_2-$), 6.75–7.3 (m, 7H at C-3, C-5, C-3′, C-5′, C-2, C-6 and C-6′), 7.4–7.9 (m, 4H at C-3″ and C-5″), 7.9–8.3 (m, 4H at C-2″ and C-6″); series IV (*n*-butyloxy derivative, 100 MHz) δ 0.95 (t, 6H, $2\times -CH_3$), 1.5–1.9 (m, 11H, $4\times -CH_2-$ and $-O-C-CH_3$), 4.2–4.4 (m, 6H, $3\times -OCH_2-$), 6.9–7.2

Table 2. Transition temperatures (°C) for series III and IV compounds.

n	SmC	I
Series III		
1	—	155.0
2	—	104.0
3	—	123.0
4	(34.0)[a]	110.0
5	(26.0)	85.0
6	(44.0)	88.0
7	(42.0)	64.0
8	(50.0)	69.0
10	(52.0)	89.0
12	(58.0)	76.0
14	(65.0)	83.0
16	(57.0)	90.0
Series IV		
1	—	186.0
2	—	160.0
3	—	155.0
4	(81.0)	108.0
5	(70.0)	103.0
6	(75.0)	89.0
7	(68.0)	75.0
8	(70.0)	85.0
10	(68.0)	83.0
12	(68.0)	78.0
14	(64.0)	83.0
16	(50.0)	85.0

[a] Values in parentheses indicate monotropy.

(m, 7H at C-3, C-5, C-3′, C-5′, C-2, C-6 and C-6′), 7.5–7.8 (m, 4H at C-3″ and C-5″), 7.9–8.4 (m, 4H at C-2″ and C-6″).

DSC data for series I, II and III are given in table 5.

2.3. *Chiral dopant*

The chiral dopant, (1*R*,2*S*,5*R*)-(−)-menthyl 4-*n*-dodecyloxybenzoate was synthesized by condensing equimolar quantities of 4-*n*-dodecyloxybenzoyl chloride with (1*R*,2*S*,5*R*)-(−)-menthol in dry pyridine [21]. Elemental analysis: found C 78.48, H 10.65, N 10.96; $C_{29}H_{47}O_3$ requires C 78.56, H 10.61, N 10.83%. IR (KBr) spectra: 2900, 1720 (−COO−), 1600, 1540, 1480, 1415, 1385 and 1370 ($-CH(CH_3)_2$), 1280, 1200, 1065, 840, 760 cm^{-1}.

3. Results and discussion

3.1. *Thermal properties*

All the members of the series I 4-methyl-2′-hydroxy-4′-(4-*n*-alkoxybenzoyloxy)azobenzenes and series II 4-ethoxy-3′-hydroxy-4′-(4-*n*-alkoxybenzoyloxy)azobenzenes exhibit an enantiotropic nematic phase. The plot of transition temperature against the number of carbon atoms in the alkoxy chain (figure 1) shows a steady fall in nematic–isotropic transition point and exhibits a marked odd–even effect.

Table 3. Elemental analysis for series I and II compounds.

	% Found (% Calculated)		
n	C	H	N
Series I			
1	69.48(69.61)	4.84(4.97)	7.56(7.73)
2	70.18(70.21)	5.64(5.32)	7.76(7.45)
3	70.68(70.77)	5.81(5.64)	7.03(7.18)
4	71.50(71.29)	5.84(5.94)	6.82(6.93)
5	71.68(71.77)	6.14(6.22)	6.93(6.70)
6	72.65(72.22)	6.25(6.48)	6.24(6.48)
7	73.08(72.65)	6.57(6.73)	6.41(6.28)
8	73.11(73.04)	6.84(6.96)	6.20(6.09)
10	73.63(73.77)	6.36(7.38)	5.68(5.74)
12	74.03(74.42)	7.66(7.75)	5.28(5.43)
14	75.11(75.00)	7.68(8.09)	5.09(5.15)
16	75.24(75.52)	8.67(8.39)	4.68(4.89)
Series II			
1	67.58(67.35)	5.23(5.10)	7.06(7.14)
2	67.74(67.98)	5.49(5.42)	6.95(6.89)
3	68.32(68.57)	5.84(5.71)	6.73(6.66)
4	68.98(69.12)	5.74(5.94)	6.27(6.45)
5	69.41(69.64)	5.98(6.25)	5.93(6.25)
6	69.89(70.13)	6.25(6.49)	5.88(6.06)
7	70.71(70.59)	6.90(6.72)	5.63(5.88)
8	70.91(71.02)	6.65(6.94)	5.43(5.71)
10	71.66(71.78)	7.21(7.34)	5.25(5.41)
12	72.32(72.53)	7.63(7.69)	4.96(5.13)
14	72.98(73.17)	7.82(8.01)	4.60(4.88)
16	73.53(73.75)	8.12(8.31)	4.47(4.65)

The first three homologues of both the series III 4-methyl-2′,4′-bis(4-*n*-alkoxybenzoyloxy)azobenzenes and series IV 4-ethoxy-2′,4′-bis(4-*n*-alkoxybenzoyloxy)azobenzenes are non-mesogenic. The *n*-butyloxy to *n*-hexadecyloxy derivatives exhibit a monotropic smectic C phase. The plot of transition temperature against the number of carbon atoms in the alkoxy chain (figure 2) shows a steady rise in the isotropic transition temperatures for series III, and a steady fall for series IV. Both the series exhibit an odd–even effect to the smectic–isotropic phase transitions.

Table 6 summarizes the average thermal stabilities and comparative geometry of the present series I–IV and the structurally related series A [13], B [22] and C [14]. The nematic thermal stabilities of series I and II are almost the same. This is understandable as there is little difference in the geometry of the two series, only the terminal group being changed from $-CH_3$ to $-OC_2H_5$. The common aspect between the series I, II and A is that they all have three phenyl nuclei, an ester central linkage and a lateral phenolic −OH group, though its position is on the terminal phenyl ring in series A. The difference between series I, II and A is in the central linkage. Series I and II have an azo central linkage whereas series A has an azomethine central linkage.

Table 4. Elemental analysis for series III and IV compounds.

	% Found (% Calculated)		
n	C	H	N
Series III			
1	70.48(70.16)	4.81(4.84)	5.86(5.64)
2	70.67(70.99)	5.13(5.34)	5.68(5.34)
3	71.72(71.74)	5.65(5.80)	5.21(5.07)
4	72.86(72.74)	6.22(6.21)	4.85(4.83)
5	73.20(73.03)	6.36(6.58)	4.47(4.61)
6	73.64(73.59)	6.60(6.92)	4.38(4.40)
7	74.26(74.10)	7.48(7.23)	3.99(4.22)
8	74.61(74.57)	7.40(7.51)	4.12(4.05)
10	75.26(75.40)	7.86(8.02)	4.04(3.74)
12	76.48(76.12)	8.35(8.46)	3.69(3.48)
14	77.00(76.77)	8.62(8.84)	3.57(3.26)
16	77.18(77.29)	8.96(9.17)	3.16(3.06)
Series IV			
1	68.61(68.44)	4.77(4.94)	5.58(5.32)
2	69.56(69.31)	5.53(5.42)	5.16(5.05)
3	70.34(70.10)	5.92(5.84)	4.90(4.81)
4	71.18(71.30)	6.43(6.61)	4.59(4.65)
5	71.35(71.47)	6.37(6.58)	4.28(4.39)
6	71.79(72.09)	6.74(6.87)	4.08(4.26)
7	72.76(72.62)	6.98(7.20)	3.96(4.03)
8	73.32(73.13)	7.24(7.48)	3.62(3.85)
10	74.18(74.04)	7.76(7.91)	3.46(3.59)
12	74.42(74.39)	8.19(7.96)	3.36(3.32)
14	75.26(75.51)	8.46(8.36)	3.45(3.12)
16	76.41(76.65)	8.89(8.62)	2.46(2.50)

Table 5. DSC data for series I–III compounds.

Series	*n*[a]	Transition	H/J g^{-1}	S/J g^{-1} K^{-1}
I	4	Cr–N	39.171	0.09742
		N–I	2.655	0.00507
	5	Cr–N	83.380	0.21243
		N–I	2.288	0.00441
	6	Cr–N	21.363	0.05475
		N–I	1.013	0.00196
	7	Cr–N	51.633	0.13301
		N–I	2.354	0.00471
	8	Cr–N	17.948	0.04583
		N–I	0.801	0.00163
	12	Cr–N	92.280	0.24674
		N–I	2.017	0.00441
	14	Cr–N	42.126	0.11089
		N–I	1.198	0.00267
	16	Cr–N	15.472	0.04155
		N–I	0.435	0.00098
II	14	Cr–N	48.058	0.12447
		N–I	0.683	0.00147
III	10	Cr–I	70.959	0.19977
		I–SmC	0.273	0.00085

[a] See tables 1 or 2.

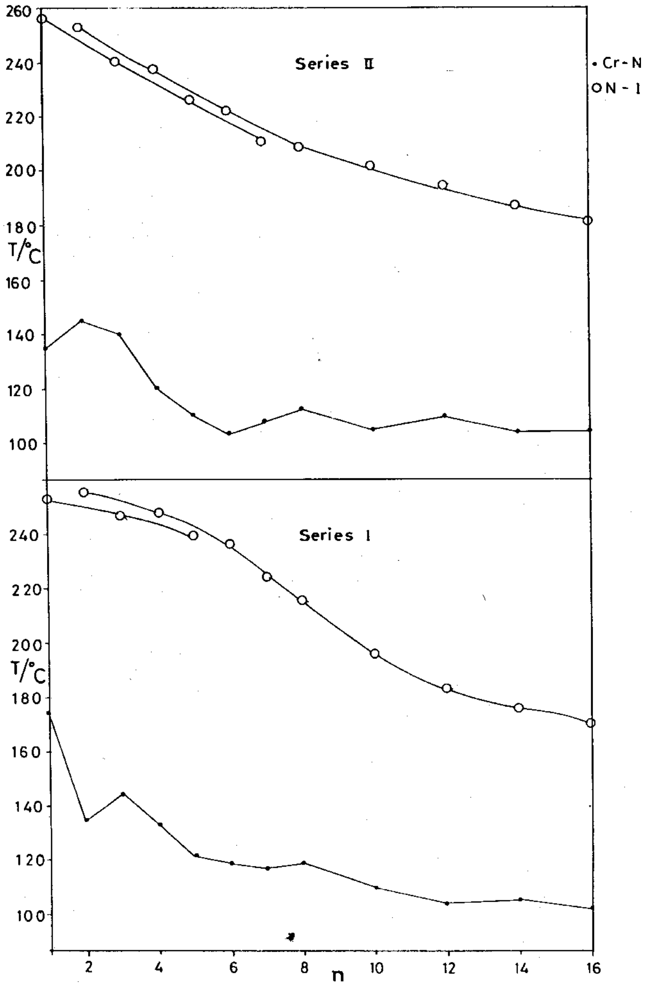
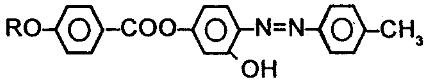


Figure 1. The phase behaviour for series I and II compounds.

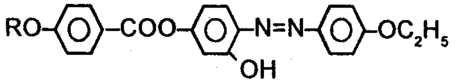
Additionally, series I and II have a terminal alkyl and alkoxy substituent whereas series A has none. The terminal group enhances overall the polarizability of the molecules, hence the thermal stabilities of series I and II should be higher than those of series A, which is the case. This indicates that the small effect of changes in the central linkage and lateral hydroxy group position is outweighed by the effect of the terminal group, as far as nematic thermal stabilities are concerned. The lateral hydroxy group on the terminal ring, and the Schiff's base linkage induce the smectic mesophase in series A. Vora and Gupta [13] have explained this behaviour in series A as based on intermolecular hydrogen bonding and close packing of molecules in layers, due to a gap arising from the overall geometry of their molecules.

Reference to table 6 indicates that the average nematic thermal stabilities of series I and II are higher than those of series B. Molecules of series I, II and B differ in their lateral group and terminal group at one end. Series I and II have $-CH_3$ or $-OC_2H_5$ terminal groups, respectively,

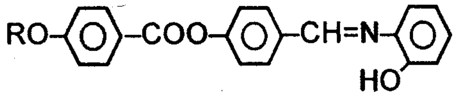
Table 6. Comparative geometry and average thermal stabilities of series I–IV, A, Band C (°C).



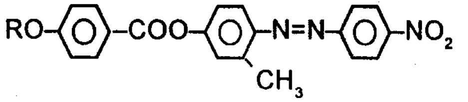
Series I



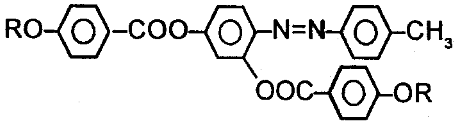
Series II



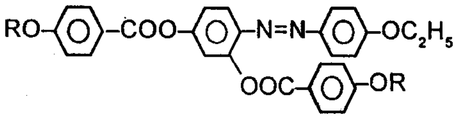
Series A



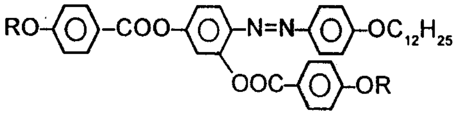
Series B



Series III



Series IV



Series C

Transition	I	II	A	B	III	IV	C
Smectic to nematic or isotropic (C_4–C_{16})	—	—	136.6	154.33 (C_{12}–C_{16})	47.5	68.2	54.05 (C_{12}–C_{16})
Nematic to isotropic (C_1–C_{14})	225.45	221.6	155.6 (C_1–C_8)	201.68	—	—	71.55 (C_4, C_8, C_{12}, C_{16})
Commencement of smectic phase	—	—	C_4	C_{12}	C_4	C_4	C_{12}

whereas molecules of series B have a terminal nitro group at the same end. Series I and II have a lateral hydroxy group whereas series B has a lateral methyl group at that position. The latter increases the breadth of the molecules leading to a decrease in mesophase thermal stabilities. In series I and II the phenolic −OH group is present *ortho* to the azo central linkage, leading to intramolecular hydrogen bonding and higher nematic thermal stabilities. No smectic mesophase is observed in either series I or II. Even though series B exhibits lower nematic thermal stabilities due to the lateral methyl group, the terminal nitro group induces the smectic mesophase in this series. Gray [11] has explained that the terminal attraction arising from the polarity of the nitro group induces smectogenic tendencies.

Table 6 further shows that the average smectic thermal stabilities of series III are lower than those of series IV and C. Molecules of series III, IV and C differ only in their terminal groups. Alkoxy groups induce a much higher polarizability in the aromatic system than do methyl groups, leading to the higher thermal stabilities of series IV and C.

The *n*-dodecyloxy terminal group enhances smectogenic tendency in a mesogenic system of rod-shaped molecules, due to enhanced lateral attractions as compared with terminal attractions [11]. Comparison of the *n*-dodecyloxy and *n*-hexadecyloxy derivatives of two series indicates that smectic thermal stabilities do not much differ. However the occurrence of nematic phases in series C compared to none in series IV cannot be

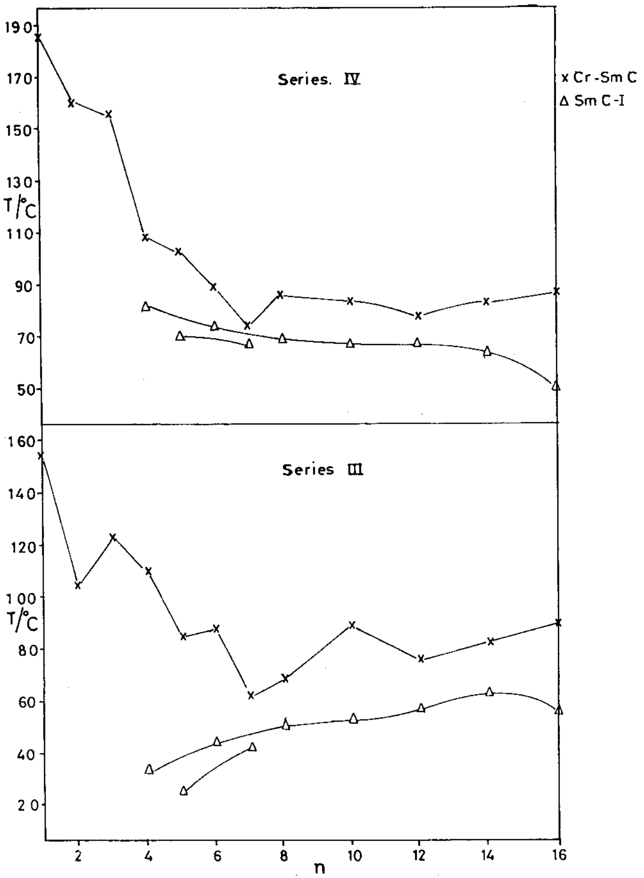


Figure 2. The phase behaviour for series III and IV compounds.

ascribed to rod-shaped molecules; the effect of trisubstituents must be significant. As mentioned above, when in a dodecyloxy derivative the terminal substituents have weaker end to end cohesive forces, the effect of the other two *n*-alkoxy substituents must predominate in the induction of the nematic phase in the system.

In the present study many low melting mesogens exhibiting smectic C phase have been synthesized; the present study facilitates the design of non-linear optical materials.

3.2. *Ferroelectric measurements*

Electro-optic and dielectric measurement of 4-methyl-2′,4′-bis(4-*n*-decyloxybenzoyloxy)azobenzene doped with 10% by weight of chiral dopant (1*R*,2*S*,5*R*)-(−)-menthyl 4-*n*-dodecyloxybenzoate (Cr 62.0°C I) were carried out. The mixture gives the monotropic phase around room temperature; transition temperatures (°C) of the mixture are: Cr (36.0) SmC* 68.0 I. The measurements were carried out in a 7.5 μm thick sample sandwiched between two conducting indium tin oxide coated glass substrates (Lucid, UK). The substrates were pre-treated with a polyimide coating. The cells were filled by capillary action with the isotropic phase of the liquid crystal. The sample was then cooled into the liquid crystal phase at 0.1° min^{-1} in a Linkam TP90 and THS600 temperature programmer having a hot stage. The thermal polarizing microscopy of the sample was observed using a Getner polarizing microscope.

3.2.1. *Polarization measurements*

A Sawyer–Tower circuit was used to determine whether the material shows a hysteresis loop, i.e. spontaneous polarization. It was found that at 20, 22, 26 and 45°C, the sample shows an elliptical loop, suggesting the absence of spontaneous polarization.

3.2.2. *Dielectric measurements*

To verify the electro-optic results dielectric measurements were made using a Hewlett–Packard impedance analyser HP 4192A in the frequency range 10 Hz to 10 MHz at zero bias. The cell was calibrated using air and benzene as standard references. Figure 3 (I), a plot of dielectric constant (real ε' and complex ε'') vs. frequency at a temperature of 26°C, shows that the relaxation frequency of the molecules is $\sim$300 kHz, i.e. the molecules relax at a very high frequency; the relaxation frequency of FLCs at a temperature less than T_c^* lies in the Hz region.

It was observed that with increase of temperature ε' remains constant; see figure 3 (II), a plot of ε' vs. temperature at various frequencies 0.1–10 kHz. In the case of FLCs ε' should fall exponentially with the rise of temperature up to T_c^*, and then remain constant, contradicting these results. This shows the existence of a non-ferroelectric phase.

Figure 3 (III), a plot of ε' vs. ε'' at 26°C (a Cole–Cole plot) shows that the molecules have a single relaxation time with frequency of $\sim$300 kHz. Figure 3 (IV), shows that the molecules have again a single relaxation time at 30°C.

4. Conclusion

The mesogenic series undertest having a lateral hydroxy group exhibit nematic mesophases due to intermolecular hydrogen bonding. The esterification of this phenolic group by 4-*n*-alkoxybenzoyl chlorides has resulted in trisubstituted mesogens having a long lateral aromatic linkage. This new type of structure induces low melting smectic mesophases.

Ferroelectric measurements indicate that the doped mesogens do not exhibit spontaneous polarization, hence they cannot be used as ferroelectric materials.

The authors thank Prof. S. G. Shah and Prof. Uma Chudasama, Department of Applied Chemistry for providing research facilities.

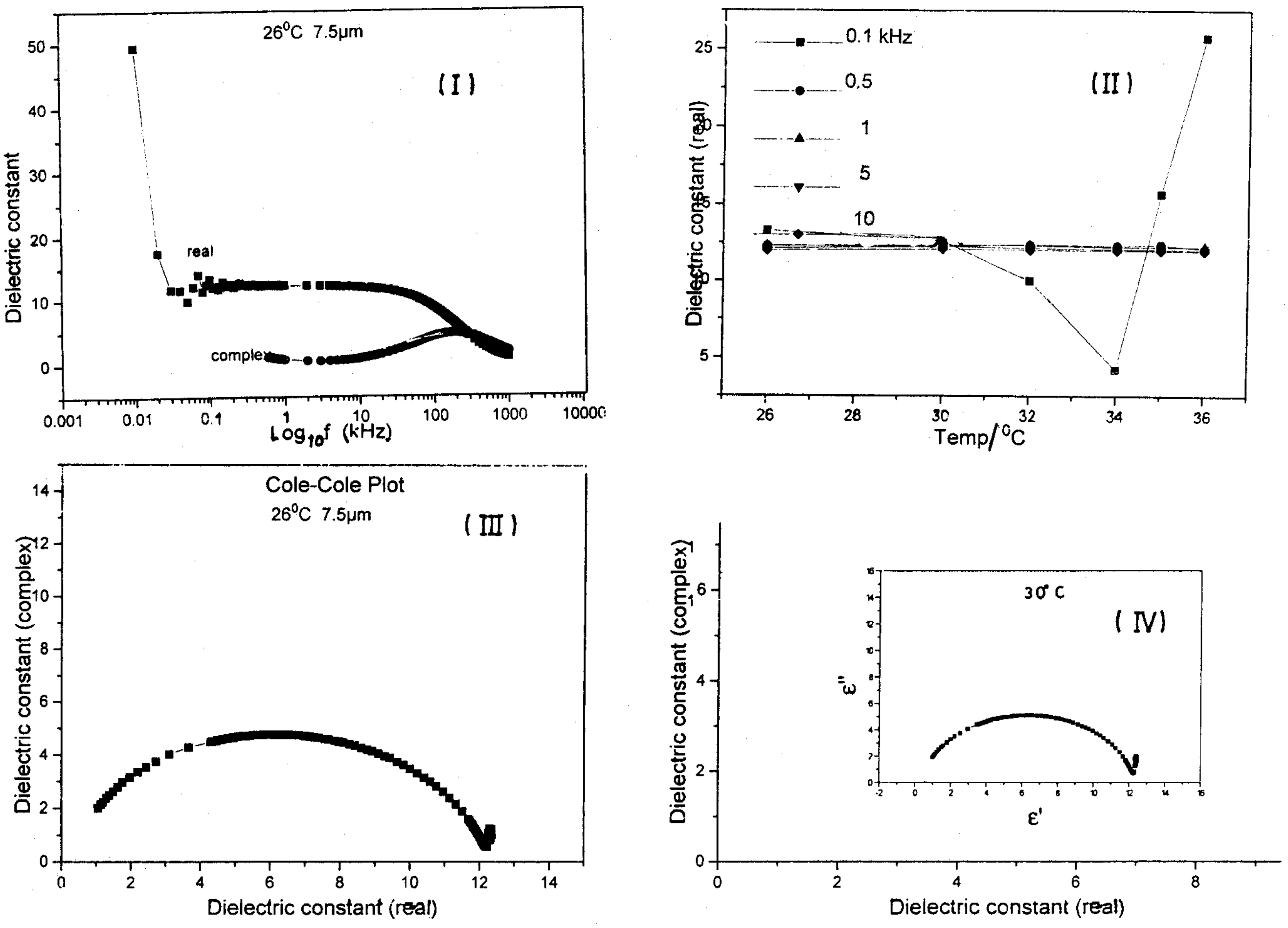


Figure 3. Dielectric measurements: for details see text.

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