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Azomesogens containing a β-chloroethyl terminal chain: synthesis and characterisation

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Azomesogens containing a β-chloroethyl terminal chain: synthesis and characterisation

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In this paper, we present the synthesis and characterisation of two new mesogenic homologous series, β-chloroethyl 4-(4′-*n*-alkoxyphenylazo) benzoates (**I**) and the β-chloroethyl [4-(4′-*n*-alkoxybenzoyloxy)phenylazo]-4”-benzoates (**II**), containing a terminal β-chloro ethyl chain. Series **I** was synthesised by alkylation of β-chloroethyl 4-(4′-hydroxy phenylazo) benzoate with an appropriate alkyl halide, whereas series **II** was synthesised by esterification of β-chloroethyl 4-(4′-hydroxyphenylazo) benzoate with an appropriate 4-*n*-alkoxybenzoic acid. The molecular structures of these new compounds of both the series were characterised by combination of element analysis and a standard spectroscopic method. The mesomorphic behaviour was studied mainly by use of a polarised microscope and, in some cases, differential scanning calorimeter as well. In series **I**, all nine members synthesised exhibited only an enantiotropic smectic A mesophase. In series **II**, all 12 homologues exhibited an enantiotropic nematic mesophase; a smectic A phase appeared in the *n*-octyloxy derivative as an enantiotropic phase and persisted through to the *n*-hexadecyloxy member. The mesomorphic properties of both series were compared with each other and also with the properties of other structurally related series to evaluate the effects of the β-chloroethyl tail on mesomorphism.

Keywords: azomesogens; β-chloroethyl tail; smectic A; nematic

1. Introduction

Terminal groups present in a molecule have their own importance because of their polarity. It has been found that terminally substituted compounds exhibit more stable mesophases compared with unsubstituted mesogenic compounds. Any terminal group, which increases molecular polarisability without increasing molecular breadth too much, increases the thermal stability of the resulting mesophase. There are different terminal substituents employed that are either a small polar substituent (i.e. –CN or –X) or alkyl or alkoxy chains. It is well known that at least one alkyl or alkoxy chain is necessary for the molecular orientation necessary for liquid-crystal phase generation (*1–4*). A number of such mesogenic homologous esters having normal (*5–17*) and branched (*18–22*) terminal alkyl chains have been reported. However, there are very few mesogenic homologous series with a substituted ethyl tail. Weygand *et al.* (*23*) have studied an alkyl chain combining two ether functions as a terminal substituent, e.g. CH_3OCH_2O–. Liquid-crystalline twin mesogens comprising cholesteryl esterimide groups linked via a poly (ethylene oxide)s chain as well as a mesogenic homologous series of cholesteryl esterimides with oxyethylene and methylene tails have also appeared in the literature (*24*, *25*). Chiang *et al.* (*26*, *27*) studied the effect of ethoxyethoxyethoxy (EO) and butoxyethoxyethoxy (BO) tails. They observed that the BO-tailed liquid crystals have higher Sm C* stability than the corresponding EO-tailed products. Earlier we reported (*28*, *29*) methoxyethyl and ethoxyethyl esters of 4(4-*n*-alkoxybenzoloxy) benzoic acids as well as methoxyethyl trans-4 (4′-*n*-alkoxybenzoyloxy)-α-methyl cinnamates. All three mesogenic homologous series exhibited a smectic A mesophase at ambient temperatures. We reported (*30*) the binary systems of such compounds with room temperature smectogenic properties and with a wide range of temperature. We also synthesised (*31*) an extensive mesogenic homologous series having an ethoxyethyl tail with a three-phenyl ring with ester and azomethine central linkages, which exhibited nematic and/or smectic mesophases. Recently, we reported azomesogens containing an ethoxyethyl/methoxyethyl tail (*32*). We observed that such terminal chains adversely affect the mesophase thermal stability, but do not eliminate mesomorphism. As a continuation of our work on substituted ethyl tails, we have synthesised two new homologous series of Schiff's base ester compounds having a chloroethyl tail (*33*). Earlier, Vora and Patel (*34*) reported a mesogenic homologous series with a chloroethyl tail. In extension of our work on substituted ethyl tails, in this paper we report the synthesis of two new mesogenic homologous series of azoesters having chloroethyl tails to evaluate the effect of a polar chloroethyl tail on mesomorphism.

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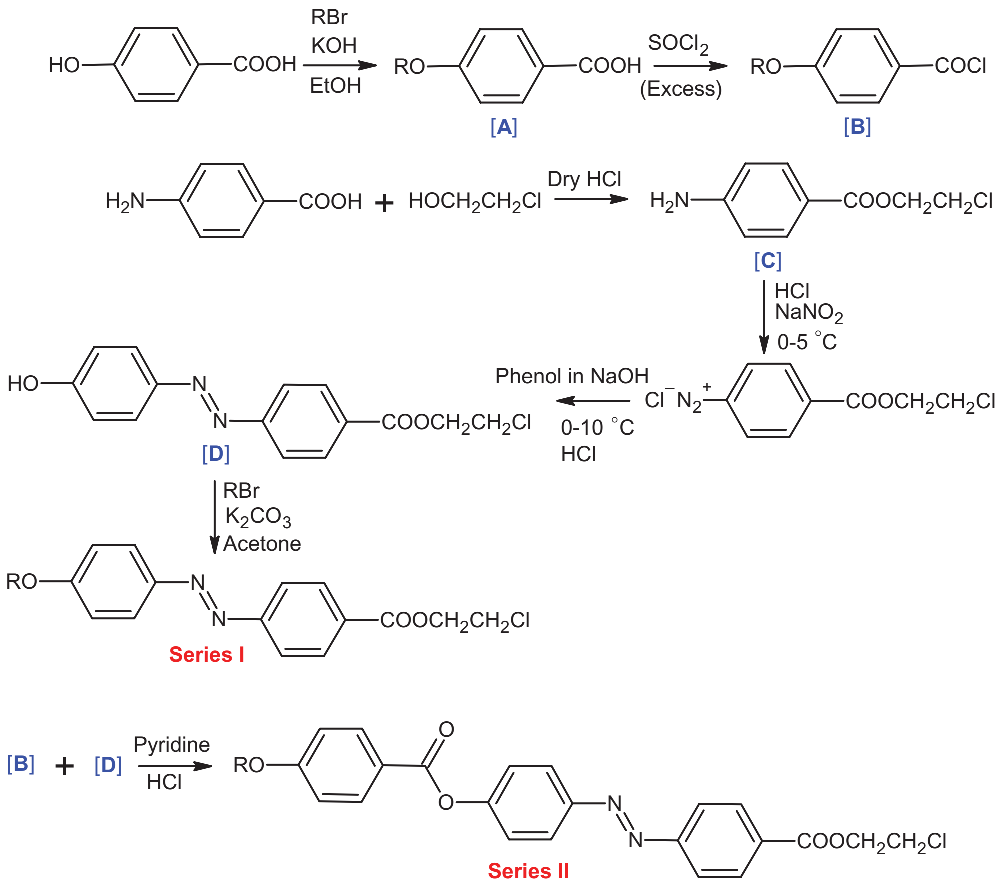
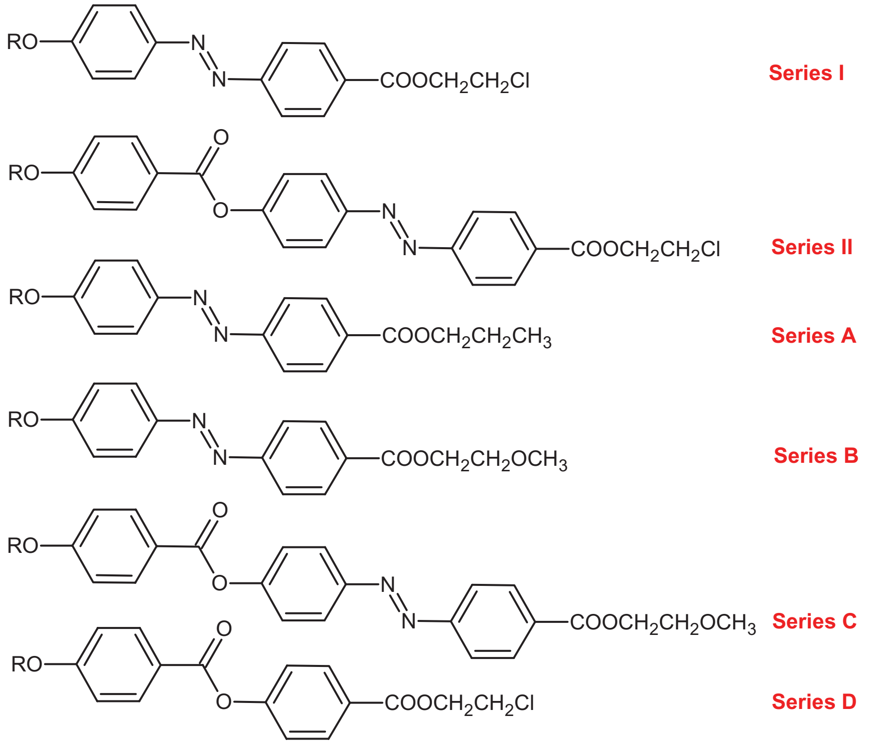
2. Experimental details

4-Hydroxybenzoic acid, the appropriate *n*-alkylhalides, 4-aminobenzoic acid, 2-chloroethanol, phenol and anhydrous K_2CO_3 were used as received. Solvents were dried and distilled before use. Microanalysis of the compounds was performed on a Coleman carbon-hydrogen analyser, and the values obtained are in close agreement with those calculated. Infrared (IR) spectra were determined for KBr pellets using a Shimadzu IR-408 spectrophotometer. ^{1}H nuclear magnetic resonance (NMR) spectra were obtained with a Perkin-Elmer R-32 spectrometer using tetramethylsilane as internal reference standard. The chemical shifts are quoted as d (parts per million) downfield from the reference; $CDCl_3$ was used as solvent for all the compounds. Liquid-crystalline properties were investigated on a Leitz Laborlux 12 POL microscope equipped with a heating stage. The enthalpies of transitions, reported as J g^2 1, were measured on a Mettler TA-4000 system, at a scanning rate of 5°C min^{-1}. The calorimeter was calibrated using pure indium as standard.

The synthetic route to compounds of series **I** and **II** is illustrated in Scheme **1**. 4-*n*-Alkoxybenzoic acids [**A**] and 4-*n*-alkoxybenzoyl chlorides [**B**] were synthesised by the modified method of Dave and Vora (*35*). *β*-Chloroethyl-4-aminobenzoate [**C**] was synthesised by the esterification of 4-aminobenzoic acid with 2-chloroethanol as described earlier (*31*). The ester was crystallised several times from carbon tetrachloride until a constant melting point (86°C) was obtained. Elemental analysis: found C 54.48, H 5.28, N 6.67 %, $C_9H_{10}NO_2Cl$ requires C54.27, H 5.01 and N 7.01%. The IR spectrum of the compound showed two bends for N–H stretching vibrations of free amino group at 3400 cm^{-1} and 3340 cm^{-1}. The –COO– stretching vibrations were seen at 1695 cm^{-1}. Other signals observed were at 1639 (N–H bending vibration), 1605, 1515, 1445, 1310, 1275, 1120, 840, 655(C–Cl) cm^{-1}. *β*-Chloroethyl 4-(4′-hydroxyphenylazo) benzoate [**D**] was synthesised by coupling the diazonium salt of *β*-chloroethyl 4-aminobenzoate with phenol (*36*). The crude dye was crystallised repeatedly from aqueous ethanol until a constant melting point (226°C) was obtained. Elemental analysis: found C 55.28, H 4.76, N 9.83%, $C_{13}H_{13}N_2O_3Cl$ requires C 55.56, H 4.63, N 10.0%. The IR spectrum of the compound





Scheme 1. The synthetic route to compounds of series **I** and **II**.

showed a broad peak associated with intermolecular hydrogen-bonded phenolic –OH between 3500 and 3200 cm^{-1}. The –COO– stretching vibrations were seen at 1720 cm^{-1}. Other signals observed were at 1600, 1500, 1475, 1345, 1260, 1135, 1010, 820, 665 (C–Cl) cm^{-1}.

2.1 *Synthesis of series I compounds*

β-Chloroethyl 4-(4′-n-hydroxyphenylazo) benzoate (**D**) (0.1 mol), the appropriate *n*-alkyl halide (0.15 mol) and anhydrous K_2CO_3 (0.15 mol) were added to dry acetone (60 ml). The mixture was heated under reflux using a water bath for 8 hours; the whole mass was then added to water. The solid was separated, dried and triturated by stirring for 30 minutes with 10% aqueous sodium hydroxide solution and washed with water. The insoluble product was thus separated from the reactants. Finally, the products were crystallised from ethanol until constant transition temperatures were obtained. The elemental analyses of all compounds of series **I** were found to be satisfactory (Table 1). IR (*n*-decyloxy derivative): 2920, 1725 (–COO–), 1600 (–N=N–), 1400, 1435, 1390, 1140, 1020, 845, 720 (C–Cl) cm^{-1}. ^1H NMR (*n*-decyloxy derivative, 300 MHz): δ 0.90 (t, 3H, $-CH_3$), 1.30–1.55 (m, 14H, 7 X $-CH_2-$), 1.75–1.85 (m, 2H, PhO–C–CH_2–), 3.85 (t, 2H, PhOCH_2–), 4.05 (t, 2H, $-CH_2C1$), 4.60 (t, 2H, $-COOCH_2-$), 7.00 (d, $J = 9$ Hz, 2H, ArH at C-3′ and C-5′), 7.90–8.00 (m, 4H, ArH at C-2′, C-6′, C3 and C-5), 8.15 (d, $J = 9$ Hz, 2H, ArH at C-2 and C-6).

2.2 *Synthesis of series II compounds*

β-Chloroethyl 4-(4′-*n*-hydroxyphenylazo) benzoate (**D**) (0.02 mol) was dissolved in dry pyridine (5 ml) and a cold solution of a 4-*n*-alkoxybenzoyl chloride (**B**) (0.02 mol) in dry pyridine (5 ml) was added slowly with constant stirring with ice bath cooling. The mixture was allowed to stand overnight at room temperature and was then acidified with cold 1:1 aqueous hydrochloric acid. The solid was separated, dried and triturated by stirring for 30 minutes with 10% aqueous sodium hydroxide solution and washed with water. The insoluble product was thus separated from the reactants. Finally, all the products were crystallised from acetic acid until constant transition temperatures were obtained. The elemental analyses of all compounds of series **II** were found to be satisfactory (Table 1). IR (*n*-butyloxy derivative): 2960, 1725 (–COO–), 1605 (–N=N–), 1505, 1460, 1385, 1280, 1220, 1140, 855, 765 (C–Cl) cm^{-1}. ^1H NMR (*n*-decyloxy derivative, 200 MHz): δ 0.90 (t, 3H, $-CH_3$), 1.40–1.60 (m, 2H, $-CH_2-$), 1.65–1.90 (m, 2H, $-CH_2-$), 3.85 (t, 2H, PhOCH_2–),

Table 1. Elemental analyses for series **I** and **II** compounds ($R = -C_nH_{2n+1}$).

Series	N	Formula	Percentage required (found)		
			C	H	N
I	4	$C_{19}H_{21}N_2O_3Cl$	63.25 (63.45)	5.83 (5.71)	7.77 (7.49)
	5	$C_{20}H_{23}N_2O_3Cl$	64.09 (64.27)	6.14 (6.28)	7.48 (7.61)
	6	$C_{21}H_{25}N_2O_3Cl$	64.86 (64.49)	6.44 (6.27)	7.21 (7.63)
	7	$C_{22}H_{27}N_2O_3Cl$	65.59 (65.67)	6.71 (6.94)	6.96 (7.06)
	8	$C_{23}H_{29}N_2O_3Cl$	66.27 (66.35)	6.96 (7.20)	6.72 (6.43)
	10	$C_{25}H_{33}N_2O_3Cl$	67.49 (67.70)	7.42 (7.69)	6.30 (6.35)
	12	$C_{27}H_{37}N_2O_3Cl$	68.57 (68.64)	7.83 (7.65)	5.93 (6.14)
	14	$C_{29}H_{41}N_2O_3Cl$	69.53 (69.66)	8.19 (8.37)	5.59 (5.84)
	16	$C_{31}H_{45}N_2O_3Cl$	70.39 (70.60)	8.52 (8.69)	5.30 (5.46)
II	1	$C_{23}H_{29}N_2O_5Cl$	62.94 (63.04)	6.70 (6.49)	6.39 (6.34)
	2	$C_{24}H_{31}N_2O_5Cl$	63.65 (63.47)	6.85 (6.92)	6.19 (6.46)
	3	$C_{25}H_{33}N_2O_5Cl$	64.31 (64.66)	7.07 (7.14)	6.00 (5.81)
	4	$C_{26}H_{35}N_2O_5Cl$	84.66 (84.60)	7.25 (7.55)	5.80 (5.88)
	5	$C_{27}H_{37}N_2O_5Cl$	65.52 (65.25)	7.48 (7.55)	5.66 (5.79)
	6	$C_{28}H_{39}N_2O_5Cl$	66.08 (66.10)	7.67 (7.73)	5.51 (5.64)
	7	$C_{29}H_{41}N_2O_5Cl$	66.60 (66.59)	7.85 (7.94)	5.36 (5.08)
	8	$C_{30}H_{43}N_2O_5Cl$	67.10 (67.42)	8.02 (8.43)	5.22 (5.07)
	10	$C_{32}H_{47}N_2O_5Cl$	68.02 (68.24)	8.33 (8.69)	4.96 (4.70)
	12	$C_{34}H_{51}N_2O_5Cl$	68.86 (68.71)	8.61 (8.78)	4.73 (4.94)
	14	$C_{36}H_{55}N_2O_5Cl$	69.62 (69.47)	8.86 (8.90)	4.51 (4.12)
	16	$C_{38}H_{59}N_2O_5Cl$	70.32 (70.64)	9.10 (9.42)	4.32 (4.38)

4.05 (t, 2H, – CH_2 –Cl), 4.60 (t, 2H, –COO CH_2–), 7.00 (d, J = 9 Hz, 2H, ArH at C-3′ and C-5′), 7.35 (d, J = 9 Hz, 2H, ArH at C-3 and C-5), 7.95–8.05 (m, 4H, ArH at C-2, C-6, C-3”, C-5”), 8.05–8.25 (m, 4H, ArH at C-2′, C-6′, C-2” and C-6”).

3. Results and discussion

As a preliminary investigation, the mesophases exhibited by series **I** and **II** were examined using a polarising optical microscope. Thin films of the samples were obtained by sandwiching them between a glass slide and a cover slip. All of the compounds of series **I** and **II** show mesomorphism. On cooling the isotropic liquid on an untreated slide, focal-conic textures characteristic of the smectic A phase were observed for compounds of series **I**. In series **II** compounds, on cooling the isotropic liquid small droplets appeared, which coalesced to give classical schlieren (threaded) textures characteristic of the nematic phase. On further cooling, the higher members ($N \geq 8$) showed focal-conic textures characteristic of the smectic A mesophase. As a representative member, the associated enthalpies of transition of *n*-heptyloxy derivative of series **I** and *n*-decyloxy derivative of series **II** were measured by differential scanning calorimetry. Data are recorded in Table 2. Enthalpy changes of the various transitions agree well with the existing related literature value (*37*).

Series **I**: β-chloroethyl 4-(4′-*n*-alkoxyphenylazo) benzoates. Nine compounds of the series were synthesised and their mesogenic properties evaluated. All of the compounds exhibit an enantiotropic smectic A mesophase; transition temperatures are recorded in Table 3. The plot of transition temperature against the number of carbon atoms in the alkoxy chain (Figure 1) shows a steady fall in the smectic–isotropic transition temperature with increasing chain length.

Series **II**: β-chloroethyl [4-(4′-*n*-alkoxybenzoyloxy) phenylazo]-4”-benzoates. All 12 members synthesised exhibit an enantiotropic nematic mesophase. A smectic A mesophase appears in the *n*-butyloxy derivative as an enantiotropic phase and remains in the series up to the *n*-hexadecyloxy derivative; transition temperatures are recorded in Table 3. The entire homologous series **II** exhibits mesomorphism. The plot of transition temperatures against the number of carbon atoms in the alkoxy chain (Figure 2) shows a smooth decrease in the nematic–isotropic as well as smectic–nematic transition temperature with increasing chain length.

Table 2. Differential scanning calorimetry data for series **I** and **II** compounds.

Series	N	Transition	Peak temperature (°C)	ΔH	ΔS
I	7	Cr–Sm A	76.9	12.56	0.0359
		Sm A–I	107.2	1.78	0.0047
II	10	Cr–Sm A	103.3	47.35	0.1232
		Sm A–N	196.0	0.68	0.0015
		N–I	251.8	0.98	0.0019

Table 3. Transition temperatures (°C) of the series **I** and **II** compounds.

Series	*N*	Cr		Sm A		N		I
I	4	•	84.0	•	112.0	–	–	•
	5	•	86.0	•	111.0	–	–	•
	6	•	78.0	•	109.0	–	–	•
	7	•	77.0	•	108.0	–	–	•
	8	•	76.0	•	106.0	–	–	•
	10	•	62.0	•	103.0	–	–	•
	12	•	72.0	•	102.0	–	–	•
	14	•	58.0	•	101.0	–	–	•
	16	•	91.0	•	99.0	–	–	•
II	1	•	189.0	–	–	•	297.0	•
	2	•	183.0	–	–	•	294.0	•
	3	•	176.0	–	–	•	288.0	•
	4	•	135.0	–	–	•	278.0	•
	5	•	168.0	–	–	•	267.0	•
	6	•	148.0	–	–	•	262.0	•
	7	•	135.0	–	–	•	256.0	•
	8	•	102.0	•	201.0	•	255.0	•
	10	•	105.0	•	197.0	•	253.0	•
	12	•	85.0	•	195.0	•	251.0	•
	14	•	79.0	•	192.0	•	240.0	•
	16	•	95.0	•	187.0	•	227.0	•

Table 4 summarises the average mesophase range, average thermal stabilities and molecular structures of series **I** and **II** and the structurally related series **A** (*38*), **B** (*32b*), **C** (*32b*), **D** (*33*) and **E** (*34*) reported in the literature. Table 4 shows that series I exhibits only the smectic mesophase, whereas series **II** exhibits smectic and/or nematic mesophases. Also a comparison of *n*-decyl to *n*-hexadecyl ethers in series **I** and **II** shows that the average relative mesophase length and thermal stabilities of the smectic phase in the series **II** are greater by 71.25°C and 89.75°C, respectively, compared with series **I**. The reference to molecular structure indicates that the molecules of series **II** are longer than the molecules of series **I** because of the third aromatic ring and a central ester linkage. Gray (*39*) explained that increase in the length of the molecule, as

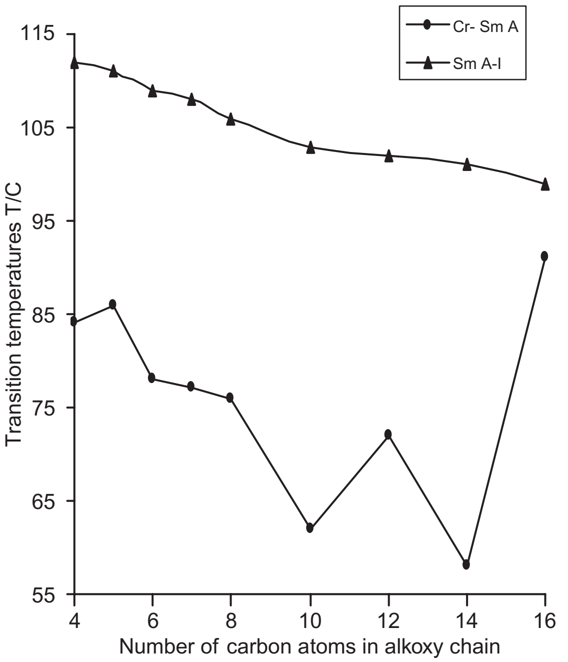


Figure 1. The phase behaviour of series **I**.

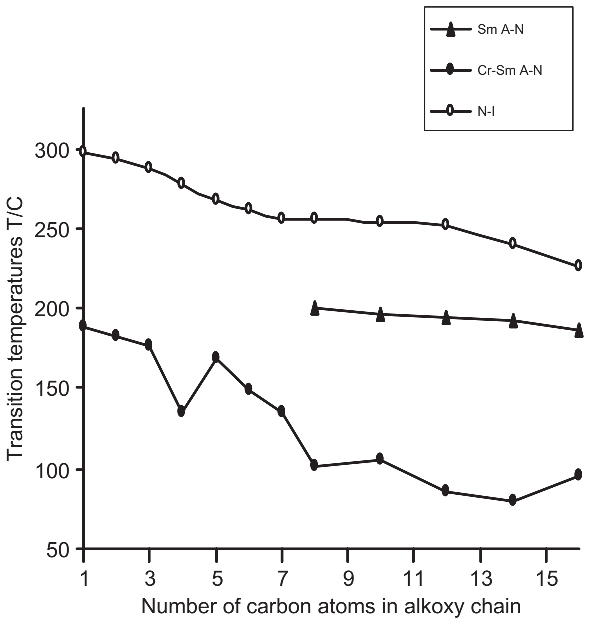


Figure 2. The phase behaviour of series **II**.

Table 4. The average mesophase length (°C) and thermal stabilities (°C).

	Mesophase length		Thermal stabilities		
Series	N (C_1–C_6)	Sm (C_{10}–C_{16})	N (C_1–C_6)	Sm (C_{10}–C_{16})	Commencement of Sm phase
I	–	30.50	–	101.25	C_4
II	114.5	101.75	281.0	191.0	C_8
A	–	12.00	–	92.50	C_4
B	–	4.20	–	78.80	C_4
C	57.00	62.75	266.8	158.25	C_{10}
D	–	21.25	–	86.75	C_6

a result of its polarisability, increases the intermolecular cohesive forces responsible for the induction of nematic mesophase as well as the wider mesophase length and the higher average smectic thermal stabilities of series **II** molecules.

Table 4 shows that the smectic mesophase range of series **I** is higher by 18.50°C and 26.30°C, respectively, compared with series **A** and **B**. It can also be noticed that the smectic thermal stabilities of series **I** are higher by 8.75°C and 22.45°C, respectively, than those of series **A** and **B**. The molecules of series **I** differ from series **A** and **B** only at the terminus. Series **I** has a chloro-terminal group, whereas series **A** has a methyl- and **B** a methoxy-terminal group instead of chloro substituent. The presence of more polar terminal chloro group increases the overall polarisability of series **I** compared with series **A** and **B**, which is responsible for the higher transition temperature and greater mesophase thermal range of series **I**. This is also reflected in series **II** and series **C** with series **II** having greater nematic and smectic mesophase lengths by 57.5°C and 39°C, respectively, and higher nematic and smectic mesophase thermal stabilities by 14.2°C and 32.75°C, respectively, than series **C**.

Reference to Table 4 indicates that the average smectic mesophase temperature range, as well as the thermal stabilities of series **I**, is higher by 9.25°C and 14.5°C, respectively, than those of series **D**. Both series differ only in the central linkage. In series **I** there is an azo central linkage whereas in series **D** there is an ester central linkage. As an azo central linkage is more coplanar than an ester central linkage, it allows packing of the molecules such that the smectic mesophase length and thermal stabilities of series **I** become higher than those of series **D**.

4. Conclusion

In this paper, we have presented the synthesis and characterisation of two new mesogenic homologous series of azobenzene derivatives containing ethoxyethyl tails, as azobenzene derivatives are more stable compared with Schiff's base derivatives. Series **I** is purely smectogenic as it is a short two-phenyl rings system, whereas series **II** exhibit nematic as well as smectic A mesophases due to the presence of an additional phenyl ring together with an ester linkage. The present series are also thermally more stable due to the presence of a more polar chloro-terminal chain compared with the structurally related compounds having less polar methoxy and a methyl tail.

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