MESOGENIC SCHIFF'S BASE ESTER WITH BUTOXY ETHYL TAIL

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ABSTRACT
New mesogenic homologous series with broken alkoxy chain, butoxy ethyl 4(4'-*n*-alkoxybenzoyloxybenzylidene) 4'-aminobenzoates has been synthesized by condensing an appropriate 4-*n*-alkoxy benzyloxy benzaldehyde with butoxy ethyl 4-amino benzoate. The synthesized compounds were characterized by combination of elemental analysis and standard spectroscopic methods.

Methoxy derivative is purely nematogenic. Ethoxy to *n*-hexyloxy derivatives exhibit enantiotropic smetic-A as well as nematic mesophases. n-heptyloxy to *n*-hexadecyloxy derivatives exhibit enantiotropic smatic-A mesophase.

The thermal stabilities and the commencement of smectic phase of those of other structurally related series.

Key words: Homologous series, broken alkoxy chain, enantiotropic, butoxy ethyl tail.

INTRODUCTION
Many liquid crystals are reported in the literature [1-3] However there is great upheaval in the synthesis of liquid crystals from two aspects. One type of synthesis is directed towards synthesis of liquid crystals which can find an application, whereas another direction is to widen the horizon of liquid crystal chemistry by synthesizing new liquid crystals of me novelty in the structural 'aspects. Compounds having an unconventional molecular structure have been a topic of research for the last 25 years. [4,5]

Literature survey indicated that esters with broken alkoxy chain are rare. Such esters are expected to exhibit nematic phases at lower temperatures if properly designed. With this expectation, Vora and Prajapati [6,7] have synthesized few mesogenic homologous seaies of esters with broken alkoxy chain as a novel terminus at the end of long polarizable molecules. In continuation of our work on broken alkoxy chain a new series of Schiff s base esters containing ethoxyethyl terminus has been synthesized and the mesomorphic properties of the present saies are compared with those of other structurally related series.

Terminal groups present in a moiety have their own importance because of their polarity. It has been found that terminally substituted functional group exhibit more stable phase transition than do non-substituted mesogenic compounds. Any terminal functional group, which increases the molecular polarizability, without significantly increasing the molecular breadth, increases the thermal stability of the resulting different shape results in a significant change in its phase transition properties. phase transition. Phase transition behaviour of the liquid crystalline compound is principally dependent on its molecular arrangement, where a slight change in the molecular

Mesomorphic behaviour of the liquid crystalline compound is principally dependent on its molecular arrangement, where a slight change in the molecular geometry results in a significant change in its mesomorphic properties. Many mesogenic homologous series with -COO, -N=N-, -CH=N-, -

CH=CH-, -C=C-, -CONH-, etc., as their central linkages, as well as alkyl and/ or alkoxy chains as terminal group, have been reported [8,9].

The mesomorphic behavior of an organic compound is sensitive to its molecular architecture; a slight change in the molecular geometry brings about considerable change in its mesomorphic properties. The detailed study of many mesogenic homologous series has helped to evolve some general rules for the effect of chemical constitution in the nematogenic and smectogenic compounds. [10,11] Dave et al. have synthesized a number of 4-*n*-alkoxy-1-naphthylidene Schiff bases and cholesteryl naphthoates and studied their mesomorphism [12,13]. Sie et al. reported Schiff base and ester linking group based heterocyclic benzoxazole liquid crystal, they studied the compounds displayed enantiotropic smectic A phase.[14]. Yeap et al. have synthesized and studied of 1,3, 5-trisubstituted benzene based star shaped derivatives containing Schiff base and ester linking group[15]. Ha et al. and his coworkers have synthesized a series of Schiff base ethers comprising a dimethyl amino group at one terminal position and an even number of carbons at the other along the molecular axis [16].

Yellamaggad et al. reported in 2007 to synthesized six homologous series based on salicylaldimine based banana shaped mesogens derived from laterally substituted resorcinol [17]. Recently, Bhoya et al. synthesized Schiff base cinnamate based homologous series and studied the effect of ortho substituted bromo group on mesomorphism [18]. Doshi et al. reported homologous series contain ester and vinyl ester linkage group [19,20]. Previously, our research group reported various homologous series based on 4-n-alkoxy benzoates and Trans 4-n-alkoxy cinnamate derivatives linking with other chalconyl derivatives and studied the effect of tail or lateral group on mesomorphism [21-25]. Prajapati and Soman [26] reported synthesis and mesomorphic properties of coumarin derivatives with chalcone and imine linkages. Prajapati and Patel [[27] have also reported Mesogenic homologous series with a butoxyethyl terminal chain.

The general structural formula for the synthesized new homologous series of compounds in are shown in Scheme 1.
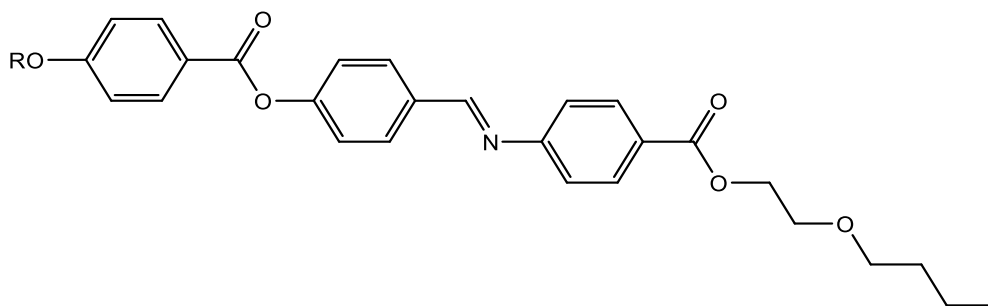
Butoxy ethyl 4(4'-*n*-alkoxybenzoyloxybenzylidene) 4'-aminobenzoates
Scheme**1**: General structural formula of series **I***n* compounds

EXPERIMENTAL

4-Hydroxybenzoic acid, the appropriate *n*-alkyl halides, 4-hydroxy benzaldehyde, 4-aminobenzoic acid and butoxy ethanol were used as received. Solvents were dried and distilled prior to use. Microanalysis of the compounds performed on a Coleman carbon- hydrogen analyser, and IR spectra were recorded on Shimadzu lR-408. NMR spectra were recorded on a Perkin-Elmer R-32 spectrometer**.** The calorimetric studies were carried out on a Mettler TA-4000 DSC apparatus by adopting a scanning rate of 5^0c/min**.** Liquid crystalline properties were investigated on a Leitz Labourlux 12POL microscope provided with a heating stage. Fluorescent *spectra* were recorded on a Shimadzu Rf540spectrophotofluromete.

4-*n*-Alkoxybenzoic acid and 4-*n*-alkoxybenzoyl chloride(A) were synthesized by the method of Dave and Vora [28]. 4-*n*-Alkoxybenzoyloxy-4 -benzaldehydes (B) were synthesized by the method of Dave

and Kurian [29]. Butoxy ethyl 4-aminobenzoate (C) was synthesized as per the method described below [30].

Dry hydrochloric acid gas was passed in mole) butoxy ethanol till it was saturated (approximately for about 3 hours). To this saturated solution 12g (0.088mole) 4-aminobenzoic acid was added and the mixture was refluxed for about 8 to 10 hours. The hot solution was then poured into excess of ice-cold water *and* sodium carbonate was added to the solution until it was neutral to litmus. The precipitated ester was filtered and dried. The product crystallized from aqueous methanol M.P. 52 ^{0}C (75-80%) The synthetic route to compounds of series **I** is illustrated in the scheme **2**.

Elemental analysis: found, C 65.92, H 8.11and N 5.95%. $C_{13}H_{19}NO_3$ Requires C 65.82%, H 8.016%and N 5.90 *%.*

The IR (KBr) spectra of the compound showed two bands for N-H stretching vibrations in the range of3300-3500 cm-$^{-1}$. The -COO- stretching vibrations were obtained *at* 1734 an-$^{-1}$. Other signals observed were *at* 2900, 1600, 1550, 1500, 1465, 1360, 1270, 1200, 1035, 830 cm^{-1}

Twelve Schiff's base esters of series I were synthesized by condensing equimolar quantities of 4-*n*-alkoxybenzoyloxy benzaldehydes and butoxyethyl-4-aminobenzoate in boiling ethanol. All the compounds of both the series were crystallized from ethanol until constant transition temperatures were obtained. [31].

The transition temperatures are recorded in Table-**I**. The elemental analysis of all the compounds are recorded in Table-**II.** The plot of transition temperatures against the number of carbon atoms in the alkoxy chain is given in figure **1.**

SPECTRAL DATA

Spectral data of *n*-butyloxy derivative of Series-I are given below.
IR (KBr)spectra: 2900, 1711(-COO-), 1610(-CH=N-), 1510, 1475, 1435,1265,1200, 1160,1075, 1010,845,755 cm^{-1}.

^{1}H NMR spectrum (400 MHz): δ 0.93 (t, 3H, -CH$_3$), 1.39-1.58 (m, 10H, 5 x -CH$_2$-), 1.60-1.84 (quant., 2H, Ar-O-C-CH$_2$-), 3.79 (t, 2H of Ar-O- CH$_2$-),0.95 (t, 3H, -CH$_3$),1.40-1.52(m,4H, 2 x-CH$_2$-), 3.2-3.51(m,4H,-CH$_2$-O-CH$_2$) 4.47 (t, 2H of -COOCH$_2$), 6.98(d, J=8.7Hz 2H,ArH),7.21-7.26(m,4H,ArH), 7.98-8.10(m,4H,ArH), 8.17(d,J=8.5Hz,2H,ArH), 8.45(s,1H,CH=N)

Spectral data of *n*-octyloxy derivative of Series-I are given below.
IR (KBr)spectra: 2900, 1712(-COO-), 1611(-CH=N-), 1510, 1475, 1435,1264,1200, 1160,1075, 1010,845,756 cm$^-$

^{1}H NMR spectrum (400 MHz): δ 0.93 (t, 3H, -CH$_3$), 1.39-1.58 (m, 10H, 5 x -CH$_2$-), 1.60-1.84 (quant., 2H, Ar-O-C-CH$_2$-), 3.79 (t, 2H of Ar-O- CH$_2$-),0.95 (t, 3H, -CH$_3$),1.40-1.52(m,4H, 2 x-CH$_2$-), 3.2-3.51(m,4H,-CH$_2$-O-CH$_2$) 4.47 (t, 2H of -COOCH$_2$), 6.98(d, J=8.7Hz 2H,ArH),7.21-7.26(m,4H,ArH), 7.98-8.10(m,4H,ArH), 8.17(d,J=8.5Hz,2H,ArH), 8.45(s,1H,CH=N)

Spectral data of *n*-dodecyloxy derivative of Series-I are given below.
IR (KBr)spectra:2900, 1712(-COO-), 1611(CH=N-), 1510, 1475, 1435,1264,1200, 1160,1075, 1010,845,756 cm^{-1}.

^{1}H NMR spectrum (400 MHz): δ 0.88 (t, 3H, -CH$_3$), 1.20-1.60 (m, 18H, 9 x -CH$_2$-), 1.70-1.80 (quant., 2H, Ar-O-C-CH$_2$-), 3.75 (t, 2H of Ar-O- CH$_2$-),0.95 (t, 3H, -CH$_3$),1.40-1.52(m,4H, 2 x-CH$_2$-), 3.2-3.51(m,4H,-CH$_2$-O-CH$_2$) 4.33 (t, 2H of -COOCH$_2$), 6.91(d, J=8.7Hz 2H,ArH),7.15-7.30(m,4H,ArH),8.7(d,J=8.5Hz,2H,ArH), 8.0-8.15(m,4H,ArH),8.42(s,1H,CH=N)

Calorimetric studies:

Calorimetry is a valuable method for the detection of phase transitions. It yields quantitative results; therefore, we may draw conclusions concerning the nature of the phases which occur during the transitions. In present study, enthalpies of derivative of series **I** (*n*=4) were measured by differential scanning calorimetry. Data are recorded in table **3.** Thermograms are given in figure **2.**

RESULTSAND DISCUSSION

Series-I: Butoxy ethyl4(4'-*n*-allroxybenzoyloxy benzylidene) 4"-aminobentoates.

Twelve members of the Series-**I** were synthesized. All the members Exhibit mesomorphism. Methoxy deriivative is purely nematogenic. Ethoxy to *n*-hexyloxy derivatives exhibit enantiotropic smectic A as well as nematic mesophases. *n*-Heptyloxy to *n*-hexadecyloxy derivatives exhibits only enantiotropic smectic A mesophases.It is clear from the plot of transition temperatures against the number of carbon atom in the alkoxy chain (Figure-**l**) that the nematic-isotropic transition temperatures exhibit the usual alterations associated with Homologous series of mesomorphic the containing alkyl chain made up of odd and even number of carbon atoms. Hence, the transition points lie on two falling curves.

Optical microscopy studies

The optical microscopic study concluded that the mesophase exhibited by compounds of series **I** as a primary investigation found consistent with the previous results. Microphotograph texture of droplet nematic mesophase (Series **I**; *n*=6) at 160 ℃ on cooling (Figure **3**.)

Table-**4** summarize thermal stability, mesophase temperature range and comparative geometry of the present Series-I and the structurally related Series **A**[32], **B**[33] and **C**[32].

Table **4** also indicates that the smectic A mesophase thermal stability of series **I**$_{12}$ is lower by 10.5°C than that of series **A**, whereas the smectic A mesophase temperature range is lower by 10.5°C than that of series **A**. As discussed earlier, the lower smectic A mesophase thermal stability is probably attributed to the increase in the length of the molecule of series **I**$_{12}$ has broken alkoxy chain (-C$_2$H$_4$OC$_4$H$_9$) at the terminus because of the extra two methylene groups which adversely affects the smectic A mesophase thermal stability as compared to series **A**. In addition, one should remember that the temperature range of the mesophase is determined partly by the Cr-SmA transition temperatures, and due to the very low Cr–SmA transition temperatures of series **I**$_{12}$ as compared to series **A**, the smectic A mesophase temperature range is also lower for series **I**$_{12.}$

Table **4** also indicates that the smectic mesophase range of series **B** is higher by 60° C. It can also be noticed that the smectic transition temperatures of series **B** are higher by 74° C, respectively, than those of Series-**I**. The molecules of series **B** and Series-**I** differ only at the terminus. series **B** has a chloro terminal group, whereas Series-**I** has an butoxy ethyl terminal group. The presence of polar terminal chloro group increases the overall polarizability of series **B** compared to Series **I**. which is responsible for the higher transition temperature and greater mesophase thermal range of **series B**.

Table **4** shows that the smectic mesophase range of series **C** is lower by 20° C. It can also be noticed that the smectic transition temperatures of series **C** are lower by 47° C, respectively, than those of Series-**I** to the molecular structure of both these compounds shows that the both the compounds differ in the number of benzene ring and central linkage. Series **I** are longer and more polarizable in comparison with series **C** due to the presence of additional azomethine central linkage and benzene moiety, which is responsible for the greater mesophase length and higher thermal stabilities of series **I**. Gray [34] has explained that the increase in the length of the molecules**,** as a result of its polarizability, increases the intramolecular cohesive forces which would be responsible for induction of nematic mesophase and the higher smectic thermal stabilities of the present series-**I**.

CONCLUSION

Broken alkoxy terminal chain with three phenyl rings having ester and azomethine central linkages exhibit nematic and/or smectic mesophases. The study indicated that broken alkoxy terminal chain affected more adversely on mesophase thermal stabilities as compared to branched chain terminus.

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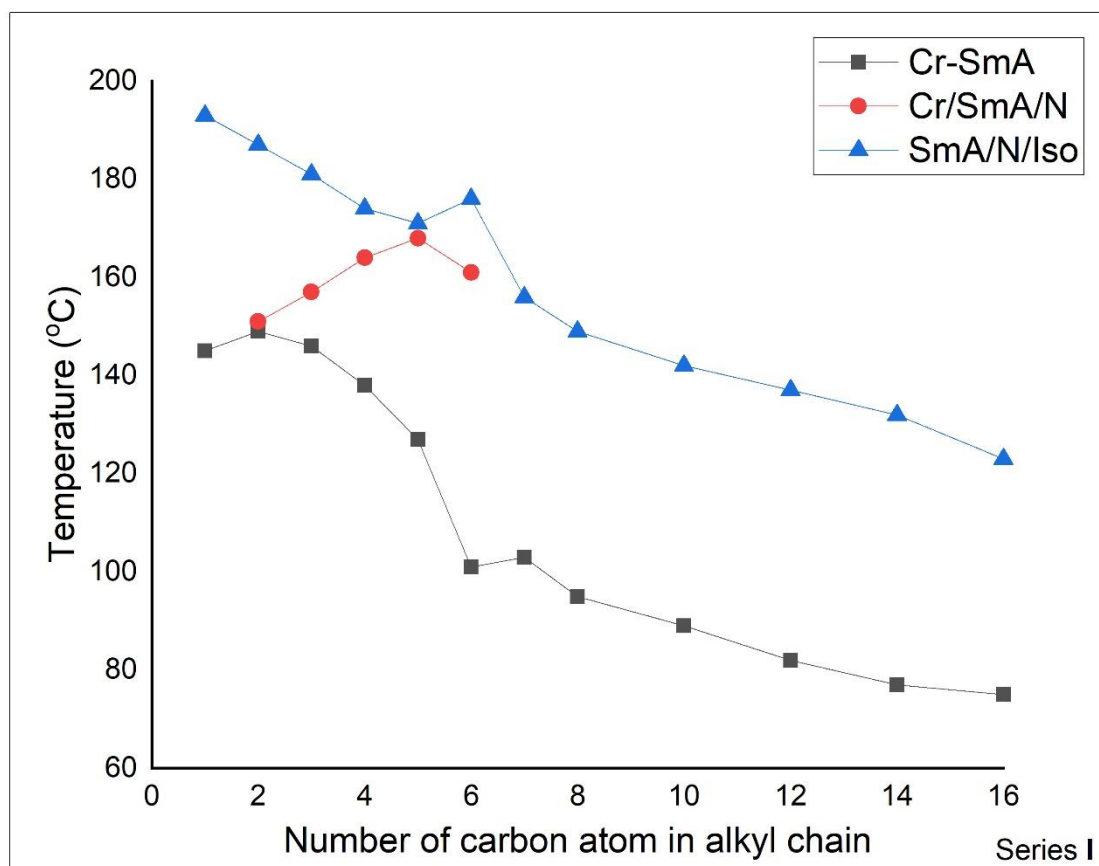
Scheme 2
Synthetic route for Series –I
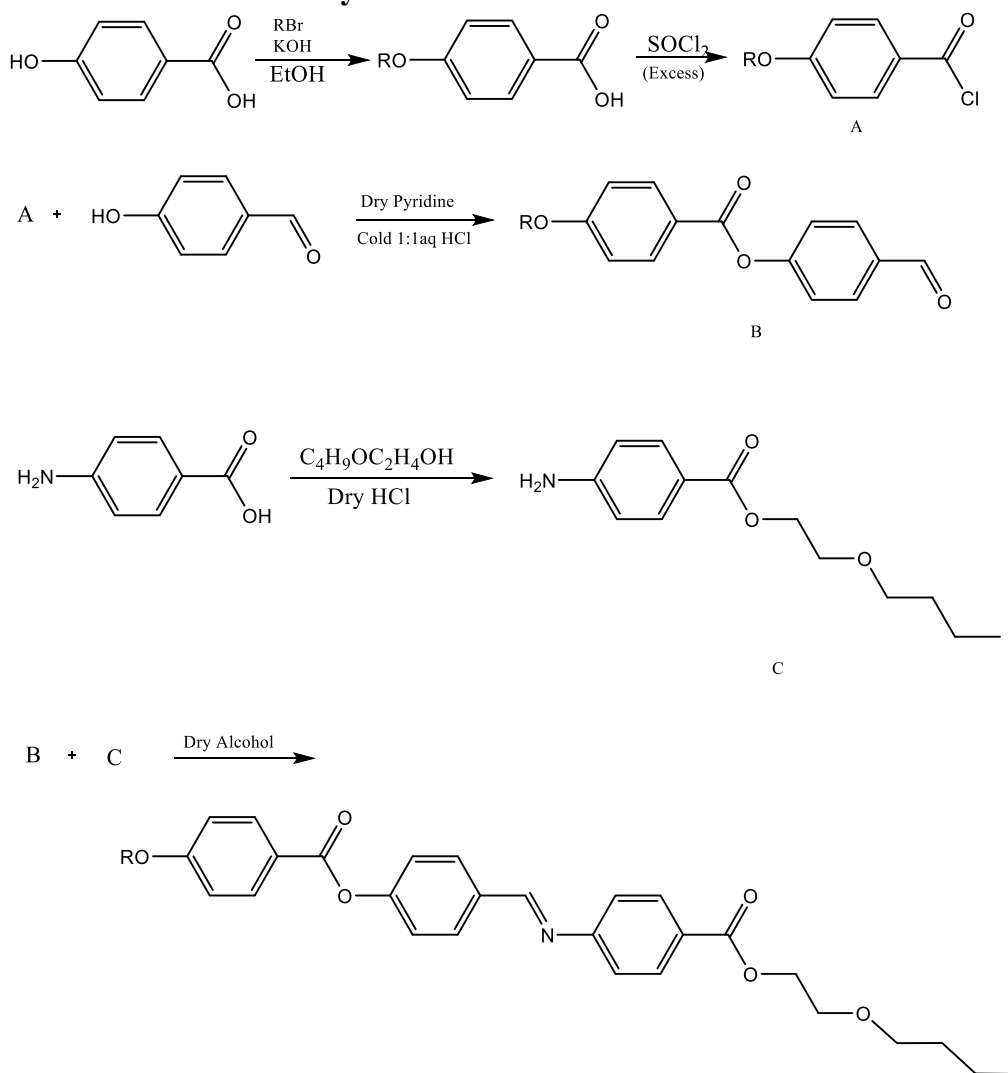
Figure 1: The phase behavior for Series **I**

Table 1: Transition temperatures (ºC) of the series I compound

R=C_nH_{2n+1}	Cr	SmA		N		I	
1	●	145			●	193	●
2	●	149	●	151	●	187	●
3	●	146	●	157	●	181	●
4	●	140	●	179	●	187	●
5	●	127	●	168	●	171	●
6	●	101	●	161	●	176	●
7	●	103	●			156	●
8	●	95	●			149	●
10	●	89	●			142	●
12	●	82	●			137	●
14	●	77	●			132	●
16	●	75	●			123	●

()= monotropic value; Cr=crystalline solid; SmA=smectic A phase; N=nematic phase; I=isotropic liquid phase; ●=phase exists.

Table: 2 Combustion analysis data

Sr.No.	R=n-alkyl chain	%Theoretically			%Observation		
		C	H	N	C	H	N
1	$C_{31}H_{35}O_6N$	71.93	6.82	2.71	71.23	6.72	2.51
2	$C_{35}H_{43}O_6N$	73.27	7.55	2.44	73.07	7.35	2.14
3	$C_{39}H_{51}O_6N$	74.37	8.16	15.24	74.16	8.05	15.44

Table 3: Enthalpy change, entropy change and normalized entropy of the series I4 by DSC

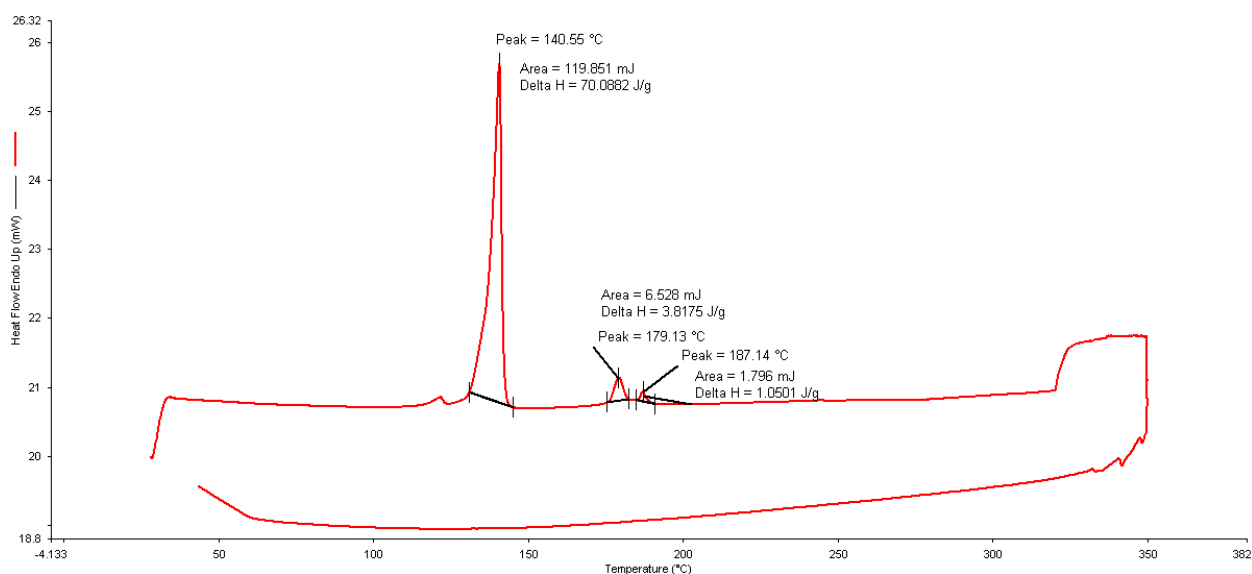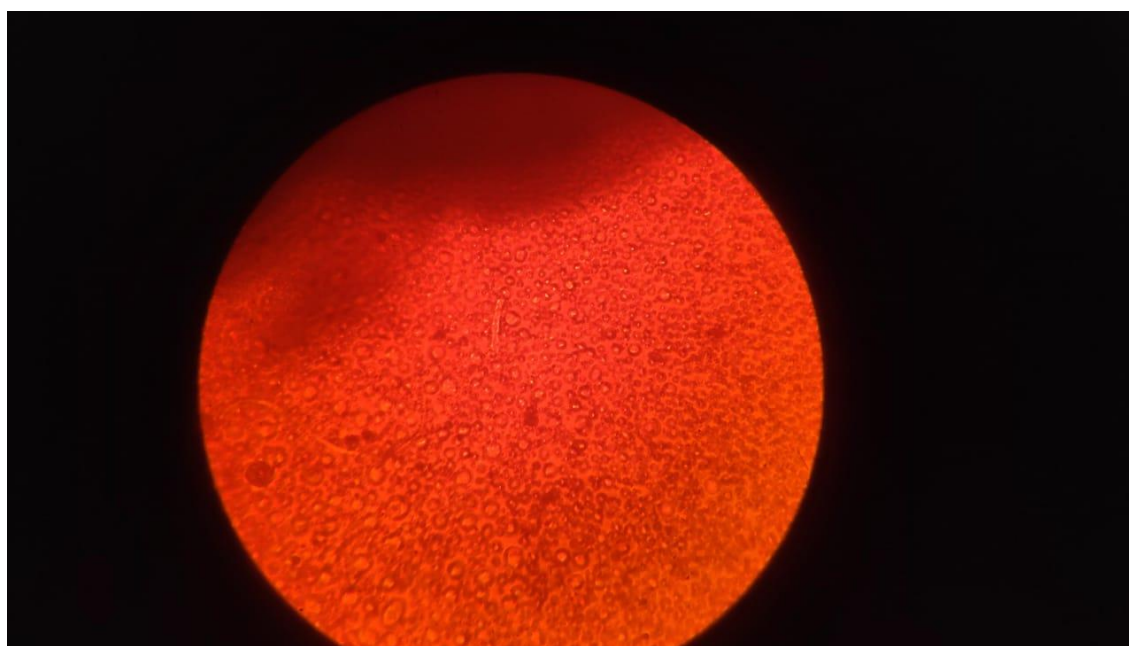| Compound	Transition	Peak temp./°C	$\Delta H/kJmol^{-1}$	$\Delta S/Jmol^{-1} K^{-1}$
I4	Cr-SmA	140.55	70.0882	0.00169
	SmA-N	179.13	3.8175	0.00857
N- I		187.14	1.0501	0.00002

Figure 2: DSC Thermogram of series **I** (*n*=**4**)

Figure 3. Microphotograph texture of droplet nematic mesophase (Series **I**; *n*=6) at 160 °C on cooling

Table:4 Comparison of the phase transition temperatures, smetic A mesophase range and comparative molecular structures of compounds **I, A** to **C**

Compound	Cr-SmA	SmA-Iso	Mesophase range/°C	Commencement of smectic A phase
I	82	137	55	C_2
A	92.5	158	65.5	C_2
B	142	211	69	C_3
C	62	70	08	C_5

Series- I

Series- A
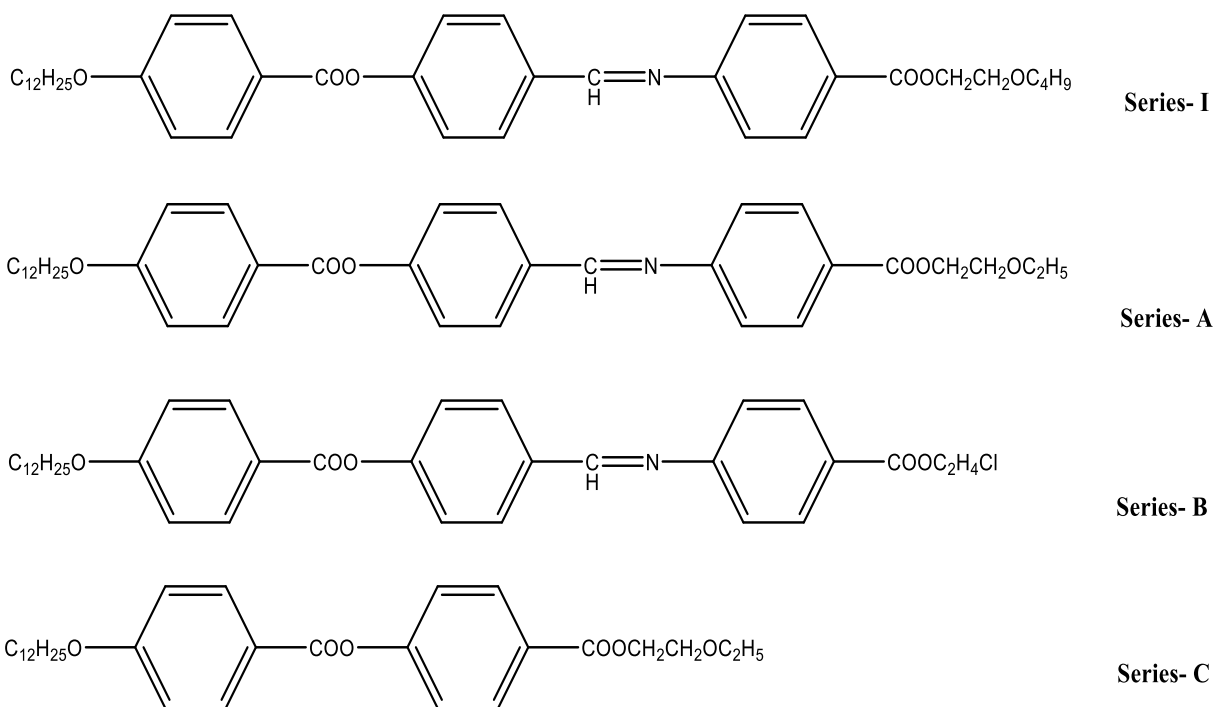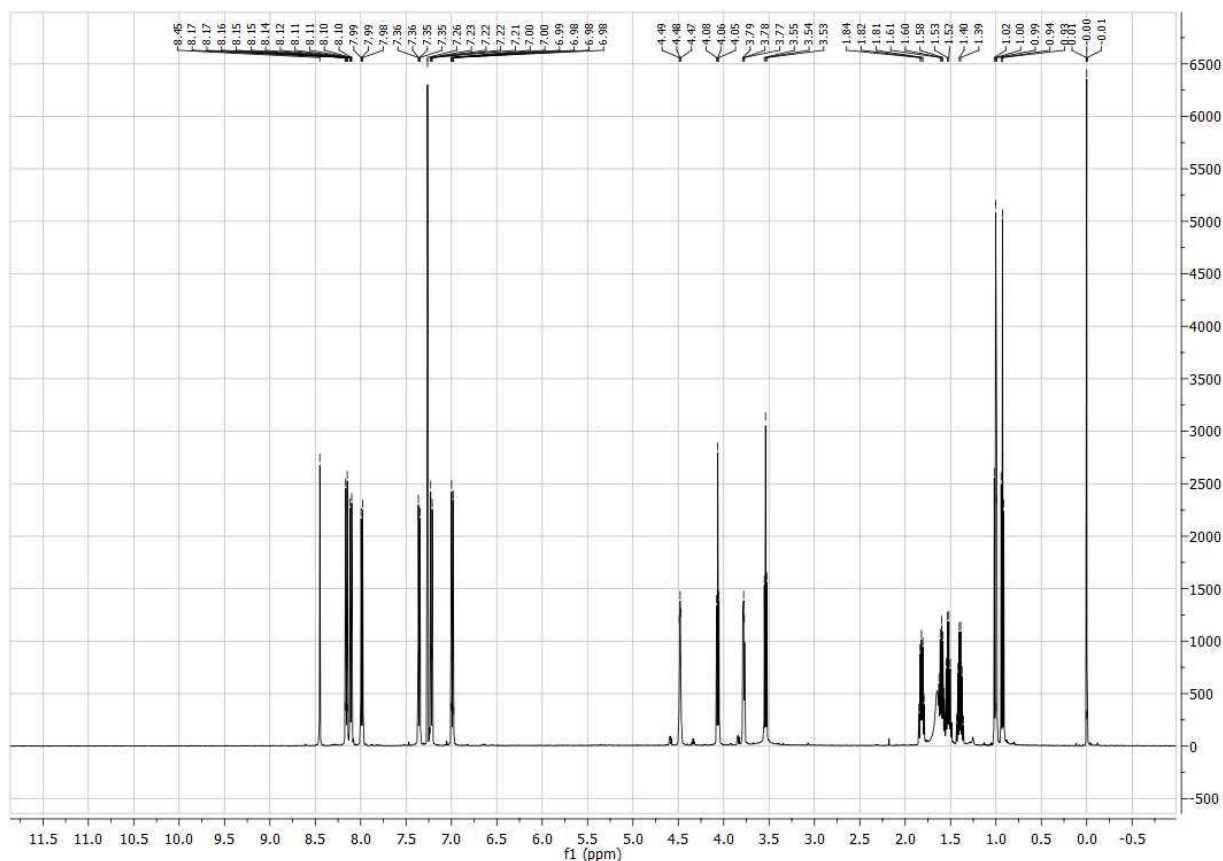
Series- B

Series- C

Figure 2: NMR data of series **I** (*n*=**4**)

^{1}H NMR spectrum (400 MHz): **δ** 0.93 (t, 3H, -CH$_3$), 1.39-1.58 (m, 10H, 5 x -CH$_2$-), 1.60-1.84 (quant., 2H, Ar-O-C-CH$_2$-), 3.79 (t, 2H of Ar-O- CH$_2$-),0.95 (t, 3H, -CH$_3$),1.40-1.52(m,4H, 2 x-CH$_2$-), 3.2-3.51(m,4H,-CH$_2$-O-CH$_2$) 4.47 (t, 2H of -COOCH$_2$), 6.98(d, J=8.7Hz 2H,ArH),7.21-7.26(m,4H,ArH), 7.98-8.10(m,4H,ArH), 8.17(d,J=8.5Hz,2H,ArH), 8.45(s,1H,CH=N).
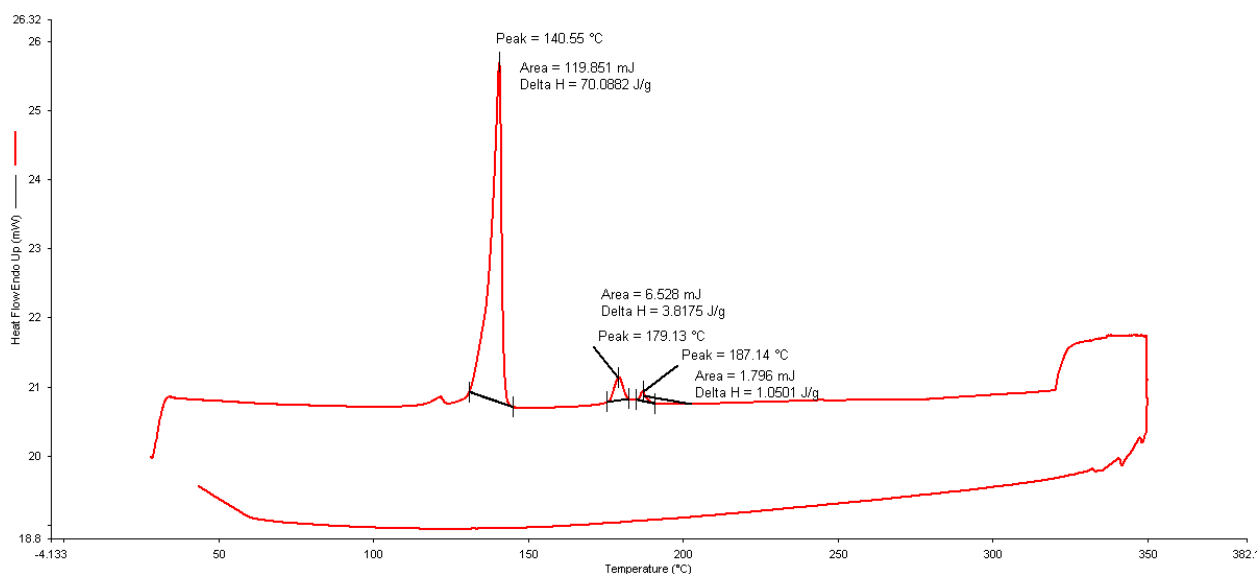

Figure 2: DSC Thermogram of series **I** (*n*=**4**)

IR (KBr)spectra: 2900, 1717(-COO-), 1601(-CH=N-), 1510**,** 1475, 1435,1265,1200, 1160,1075, 1010,845,755 cm^{-1}.

Name	Description
4ABB_1_1_1	Sample 002 By Administrator Date Tuesday, December 05 2023