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## Synthesis and Liquid Crystalline Properties of Novel Phenyl Ethylene Derivatives

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### ABSTRACT

A Novel homologous series of liquid crystals, viz,  $\alpha$ -4(4'-n-alkoxy cinnamoyloxy) benzoyl- $\beta$ -4''-pentyloxy phenyl ethylene, has been synthesized. The series consists of eleven homologs. The methyl to pentyl derivatives are not liquid crystalline and the rest of the homologs are enantiotropic liquid crystals. The decyl and dodecyl derivatives of the series are enantiotropically smectogenic in addition to nematogenic in character, but the hexyl, tetradecyl and hexadecyl homolog derivatives of the series are only nematogenic without the exhibition of a smectic phase. The transition temperatures of the novel substances were determined by optical polarizing microscopy equipped with a heating stage. A plot of transition temperatures versus the number of carbon atoms present in the n-alkoxy terminal chain represents the phase behavior of the series. An odd-even effects not observed for the nematic-isotropic transition curve. The textures of the nematic phase are of a threaded or Schlieren type and those of the smectic A and smectic C phases are typical. Analytical and spectral data agree with the molecular structures. The smectic and nematic thermal stabilities are 122 °C and 157 °C, respectively. The smectic phase commences from the decyl homolog. Smectogenic phase lengths vary from 13 °C to 15 °C and the nematogenic phase lengths vary from 11 °C to 46 °C. The series is predominantly nematogenic and partly smectogenic with considerable mesophase length and a middle-ordered melting type. The liquid crystal properties of the present series are compared with structurally similar homologous series.

**Keywords:** Liquid crystal, smectic, nematic, Mesomorphic, thermotropic

## 1. INTRODUCTION

The unique properties of the liquid crystalline state of a substance is exploited in varying fields of application in present era. There fore, it is interesting to study the relationship between molecular structure and the liquid crystal state of a substance through homologous se- rise, whose molecular rigidity and flexibility [1-3] dictate the formation of mesomorphism. Liquid crystal phases are formed as a consequence of suitable magnitudes of anisotropic intermolecular forces of attraction [4], and as a consequence of favorable molecular rigidity and/or flexibility. The novel series under investigation has a molecular structure based on three phenyl rings linked through -CH=CH- COO- and -CO -CH =CH- centrallinking groups, two terminal end groups of OR and Cl, hence the structures are linear with no lateral substituents.

## 2. EXPERIMENTAL

### 2. 1. Synthesis

#### (a) Synthesis of 4- Hydroxy Cinnamic acid

4-Hydroxy Cinnamic acid prepared by dissolving p-Hydroxy benzaldehyde (0.15 mole) and malonic acid (0.375 mole) in pyridine (50 ml) to which few drops of Piperidine was added and then heated for a rapid evolution of CO<sub>2</sub> take place. The reaction was complete by boiling the solution for 5 minutes. It was cooled and poured in to excess of water containing enough hydrochloric acid (HCl) remove excess of pyridine. The trans-p-hydroxy cinnamic acid was separated and filtered off, washed with water and re crystallized from alcohol several as white shining crystals. The fine crystals obtained were melted sharply at their reported melting points.

#### (b) Synthesis of 4-n-alkoxy Cinnamic acid [A]

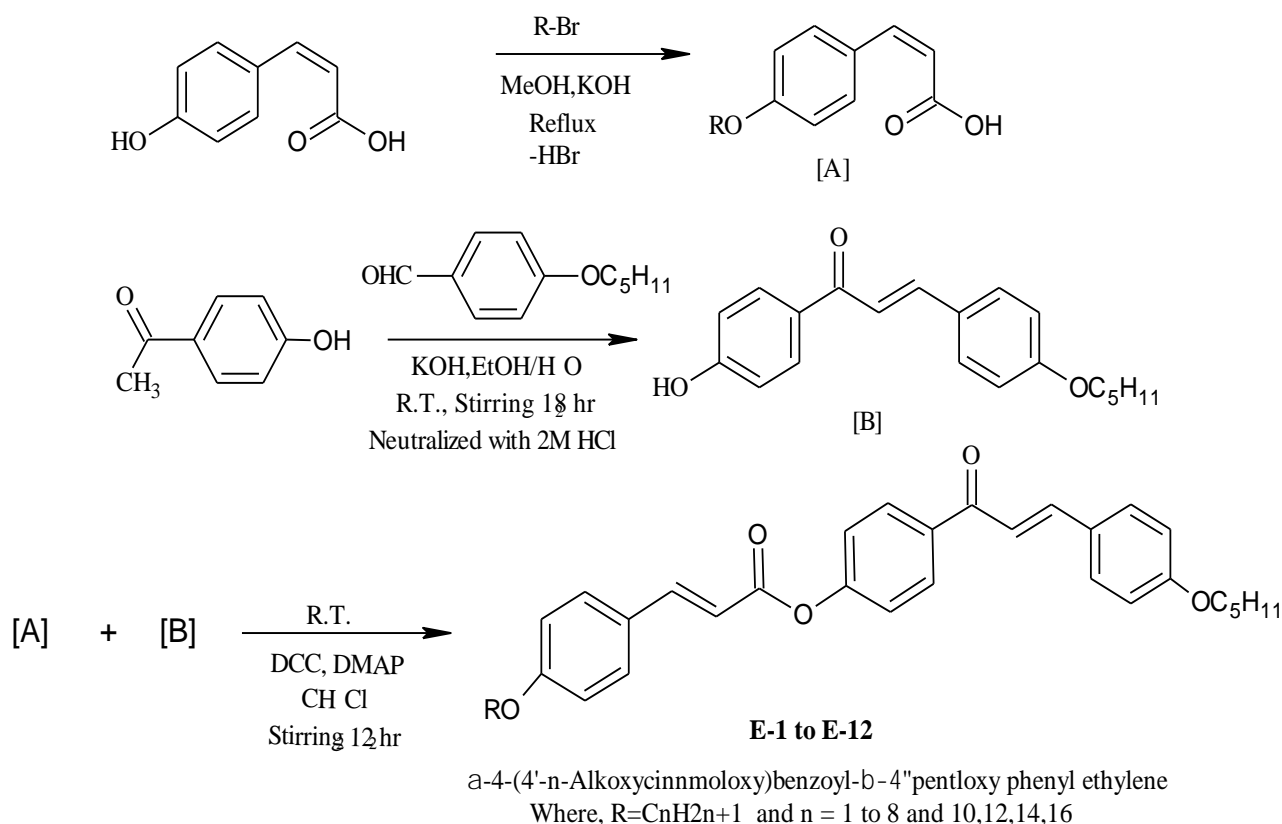
4-Hydroxy cinnamic acid (0.1 mole), corresponding alkyl halide (0.12 mole) and KOH (0.25 mole) were added dissolved in 100 ml methanol and reaction mixture was refluxed for 3 to 4 hours, 10 % aqueous KOH solution (20 ml) was added and refluxing continued for further 2 hours to hydrolyses any ester formed. The solution was cooled and acidified with 1:1 hydrochloric acid (HCl) to precipitate the corresponding alkoxy cinnamic acid. Time period for refluxing reaction mixture was extended with increasing alkyl chain length. Cinnamic acids were crystallized from ethanol (C<sub>2</sub>H<sub>5</sub>OH) or acetic acid (CH<sub>3</sub>COOH) till constant melting point were obtained. The transition temperatures are in agreement with the reported literature [5-6].

#### (c) Synthesis of $\alpha$ -4-(4'-n-Alkoxy)benzoyl- $\beta$ -4''-pentyloxy phenyl ethylene [C<sub>1</sub> to C<sub>16</sub>]

To a mixture of  $\alpha$ -4-hydroxybenzoyl- $\beta$ -4'-methylphenylethylene (0.01 mole) and 4-n-alkoxycinnamic acid (0.01 mole) in 150 ml dichloromethane, 0.001 mole of dimethyl amino pyridine (DMAP) was added. The mixture was cooled in an ice-water bath and after 10 min, 0.01 mole of Dicyclohexyl carbodiimide (DCC) was added. The mixture was stirred overnight at the room temperature. The completion of reaction was confirmed by analytical thin layer chromatography using hexane: ethyl acetate (8:2) as mobile phase. After completion of reaction, the solid mass obtained was filtered off. This crude product was crystallized from ethanol to give analytical pure product [5-6]. Analytical data support the structure and product.

## 2. 2. Characterization

Some of these homologs of the novel series were characterized for the determination of molecular structures by elemental analysis, infra red [IR] and  $^1\text{H}$  NMR (Nuclear Magnetic Resonance) techniques. Microanalysis was performed using a Perkin Elmer PE 2400 CHN analyzer. IR spectra were recorded on a Perkin-Elmer spectrum GX, and  $^1\text{H}$  NMR spectra were recorded on a Bruker instrument using  $\text{CDCl}_3$  as a solvent. The mesogenic properties and transition temperatures of the series were investigated using an optical polarizing microscope equipped with heating stage. The textures of the smectic and nematic mesophase were determined by miscibility methods.



**Scheme 1.** Synthetic route to the novel series.

## 2. 2. Analytical Data

**Table 1.** Elementary Analysis

Compound	M. F.	Elements % Found			Elements % Calculated		
		C	H	O	C	H	O
Propyl	$\text{C}_{30}\text{H}_{30}\text{O}_5$	77.17	7.05	15.78	77.19	7.03	15.76

Butyl	$C_{31}H_{32}O_5$	77.41	7.25	15.34	77.35	7.18	15.47
Pentyl	$C_{32}H_{34}O_5$	77.64	7.45	14.94	77.60	7.35	15.03

### 2. 3. Spectral Data

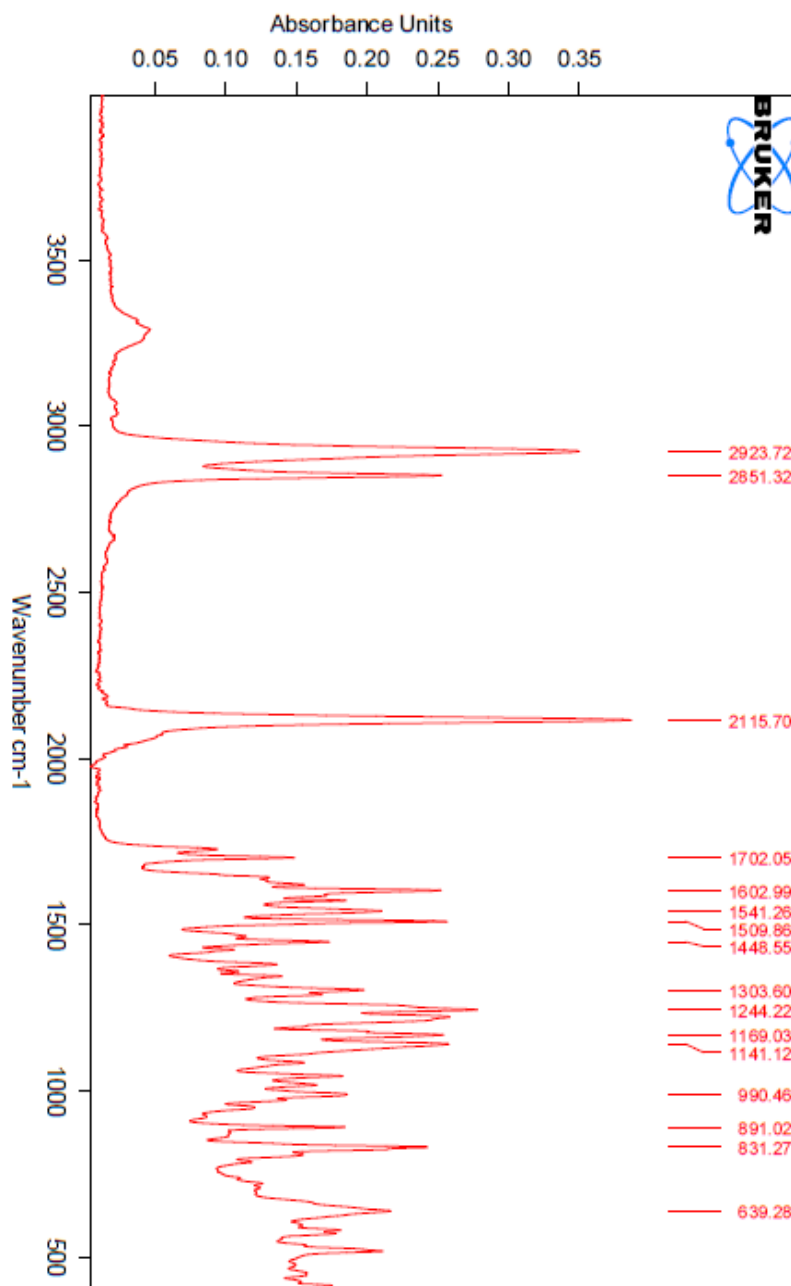
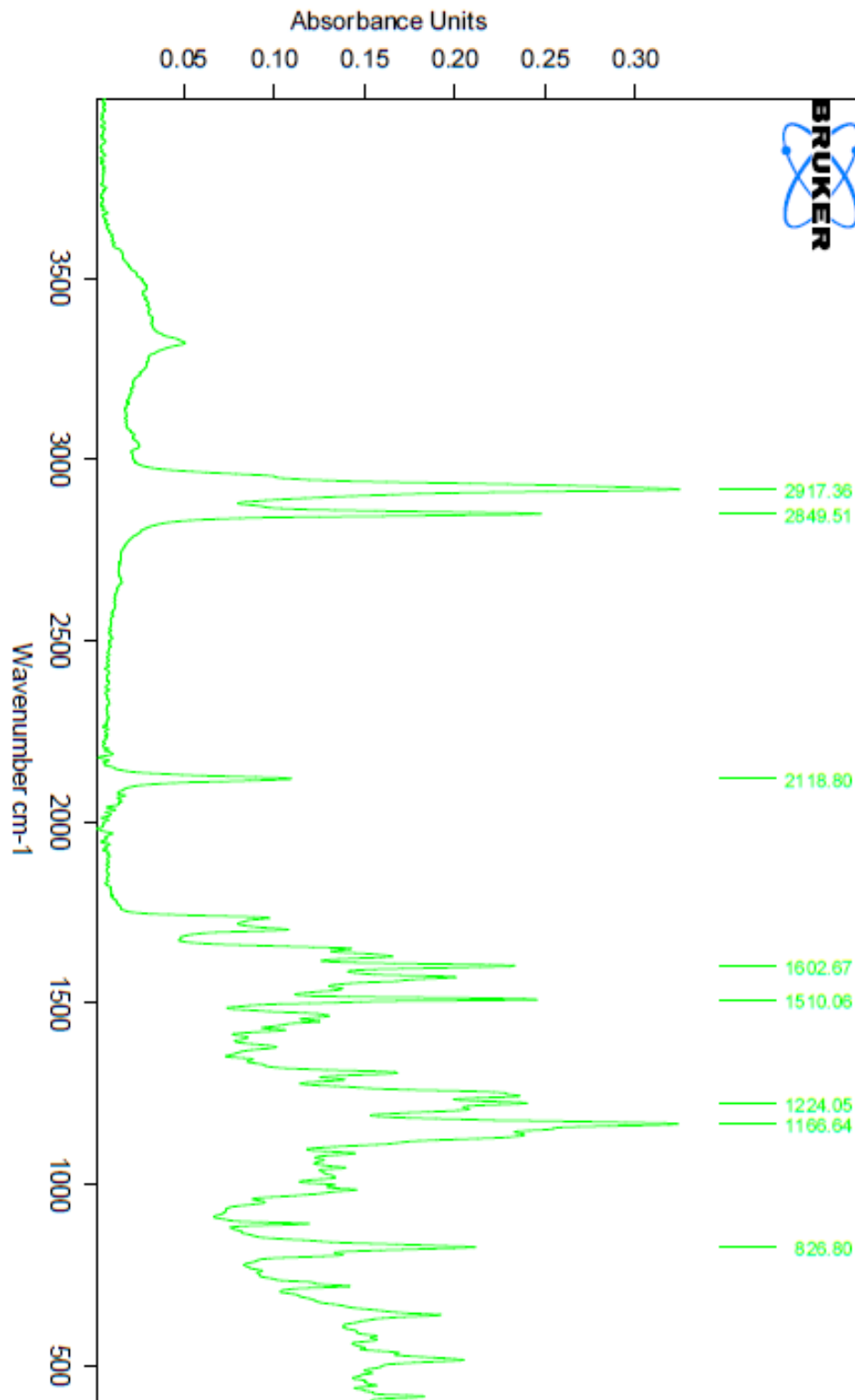


Fig. 1(A). IR Spectrum compound of C<sub>10</sub>



**Fig. 1(B).** IR Spectrum compound of C<sub>12</sub>

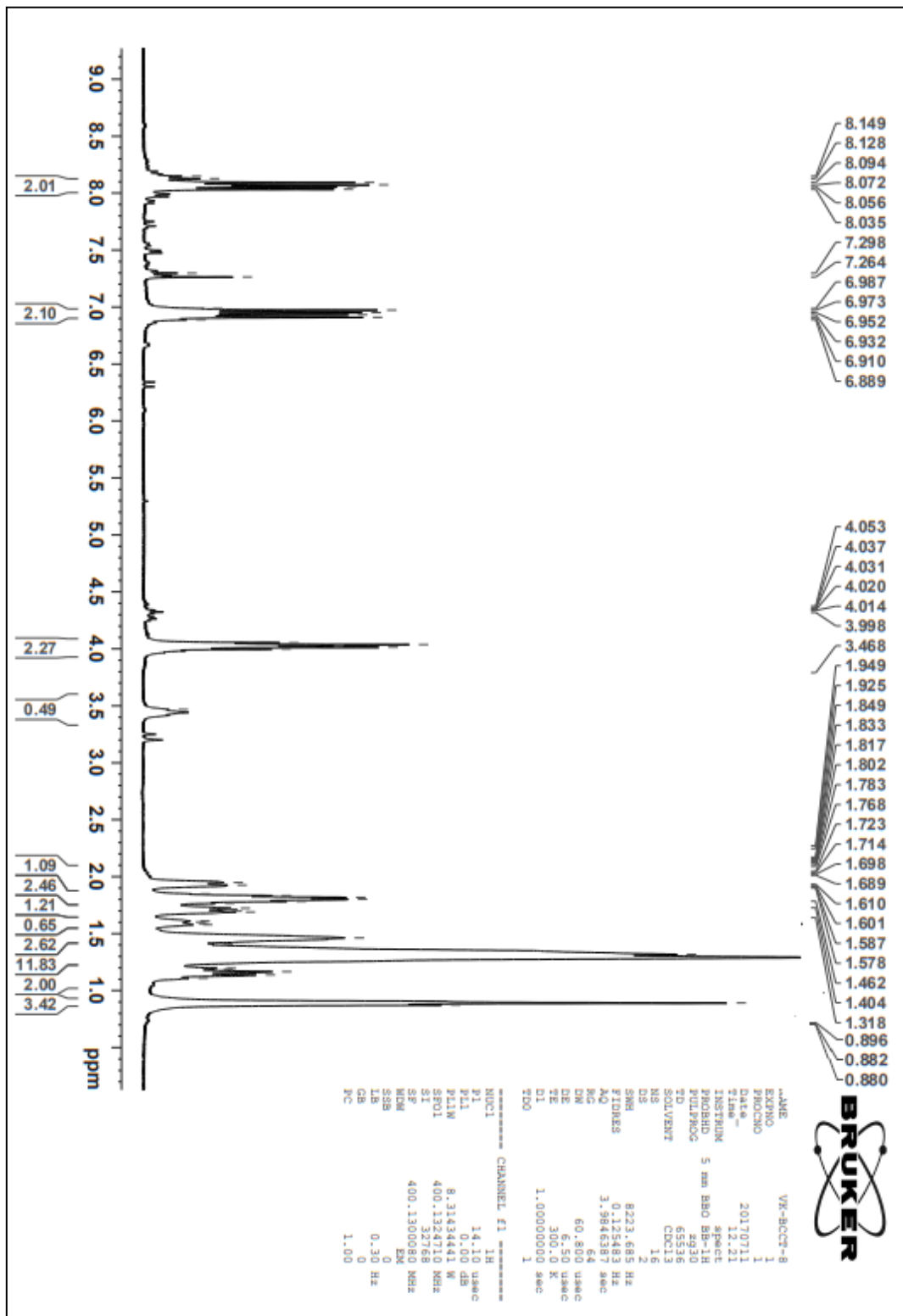


Fig. 1(C). <sup>1</sup>H NMR Spectrum compound of C<sub>8</sub>

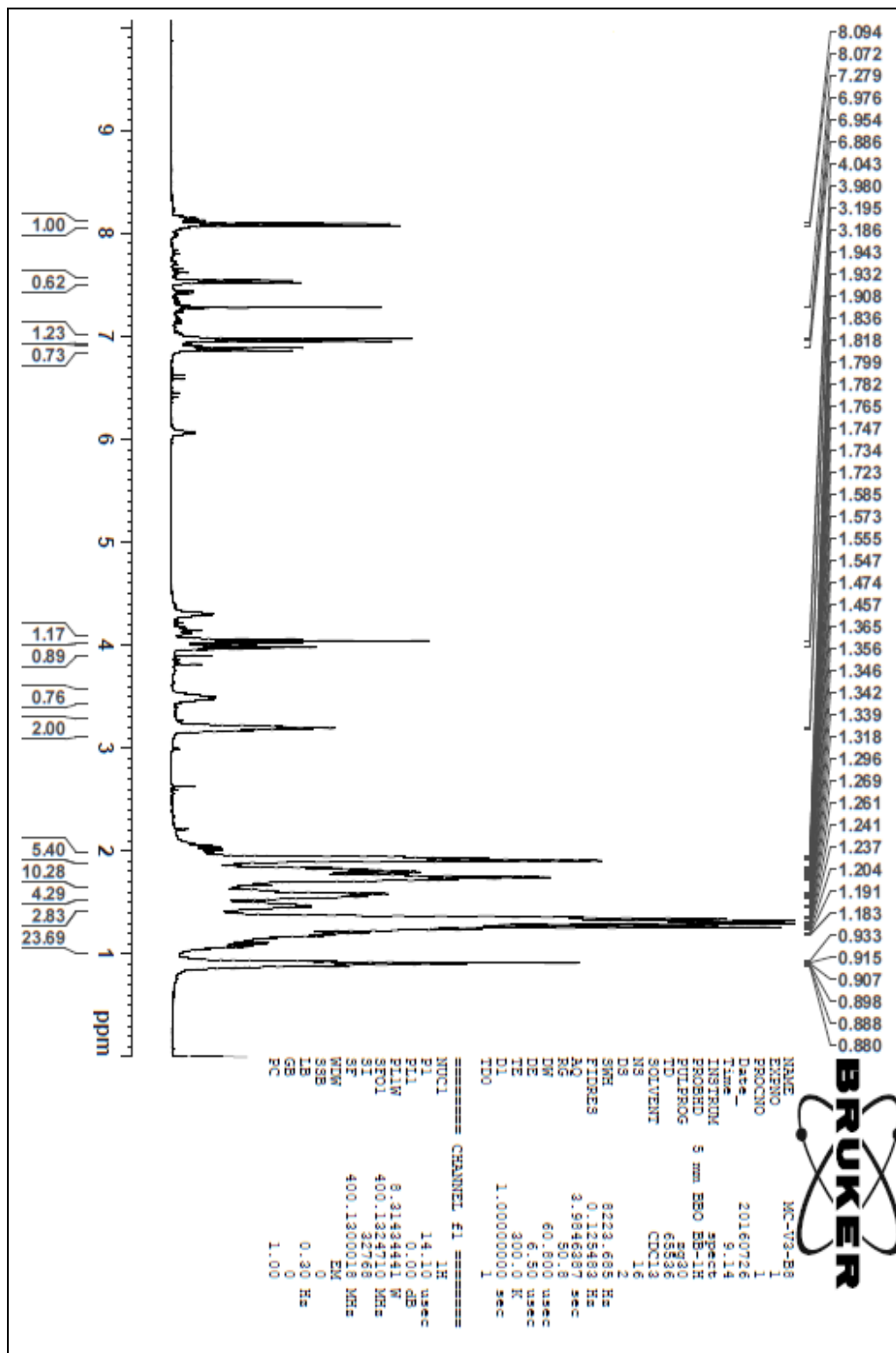


Fig. 1(D). <sup>1</sup>H NMR Spectrum compound of C<sub>12</sub>

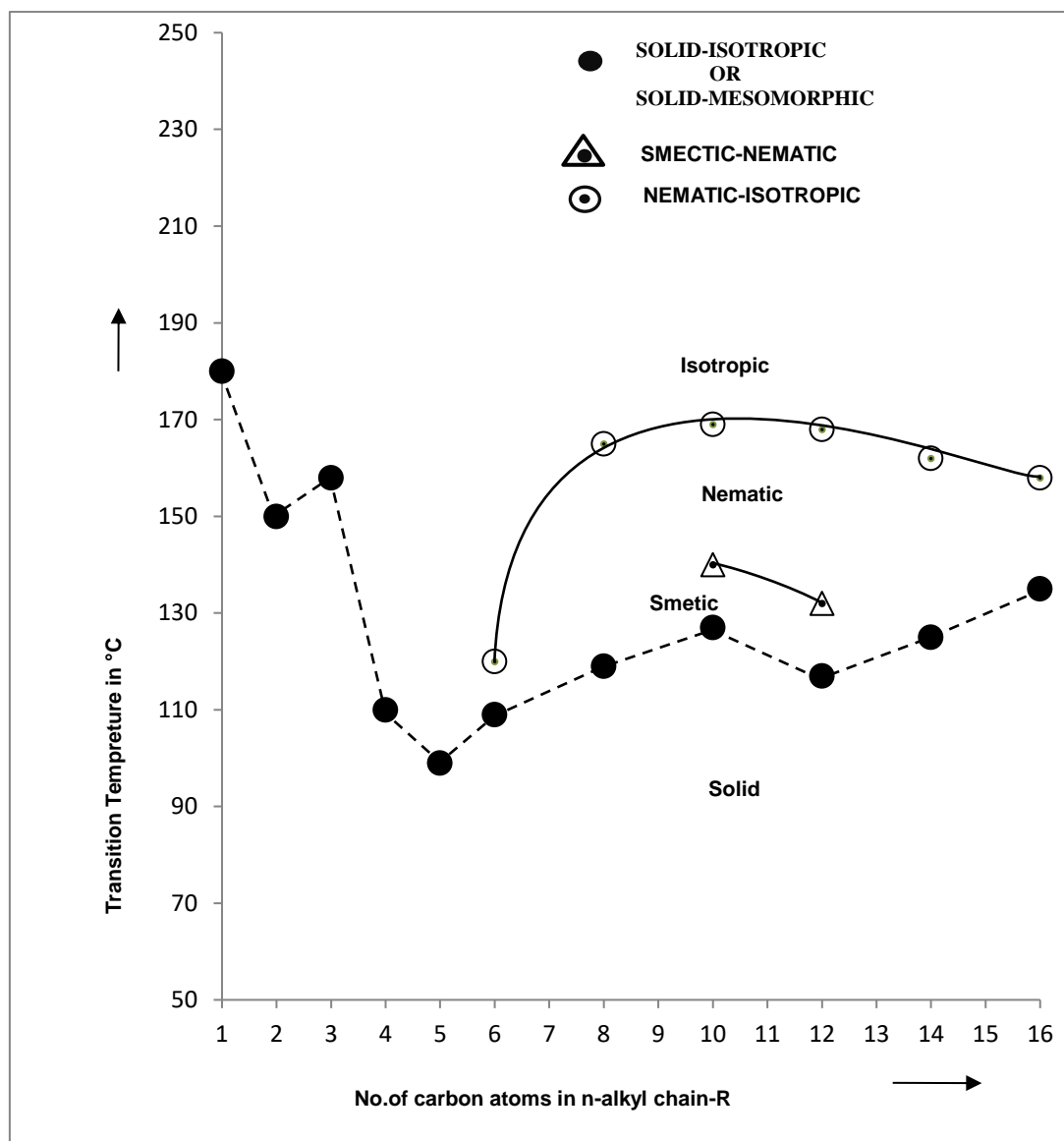


Figure 2. Phase Behavior of Series

IR (KBr) in  $\text{cm}^{-1}$  for Decyloxy homologue. ( $\text{C}_{10}$ ). 3322 (=C-H str. of phenyl nucleus), 2923 & 2851 (C-H aliphatic sym. & asym. str.), 2116 (aromatic C=O str.), 1640 (C=O str. of Chalcone), 1602 (C=C Str. of vinyl group of Chalcone & aromatic), 1541 (C-C aromatic str.), 1448 (C-H bending of alkanes), 1244-1141 (C-O-C str. of alkoxy), 1043 (C-CO-C str.) 831 (C-H bending of phenyl ring).

IR (KBr) in  $\text{cm}^{-1}$  For Dodecyloxy homologue. ( $\text{C}_{12}$ ). 3320 (=C-H str. of phenyl nucleus), 2917 & 2849 (C-H aliphatic sym. & asym. str.), 2118 (aromatic C=O str.), 1640 (C=O str. of Chalcone), 1602 (C=C Str. of vinyl group of Chalcone & aromatic), 1510 (C-C aromatic str.), 1447 (C-H bending of alkanes), 1224-1166 (C-O-C str. of alkoxy), 1050 (C-CO-C str.) 826 (C-H bending of phenyl ring).



$^1\text{H}$  NMR ( $\text{CDCl}_3$ ) in ppm for octyloxy derivative ( $\text{C}_8$ ). 0.88-0.89 (t, 3H,  $-\text{CH}_3$  of  $-\text{OC}_8\text{H}_{17}$  group), 1.12-1.27 (m, 12H,  $2\times\text{CH}_2$  of  $-\text{OC}_8\text{H}_{17}$  group), 1.31-1.61 (m, 2H, Ar-O- $(\text{CH}_2)_2\text{-CH}_2$ ), 1.68-1.94 (m, 2H, Ar-O- $(\text{CH}_2)\text{-CH}_2$ ), 3.99-4.05 (t, 2H, Ar-O- $\text{CH}_2$ ), 6.88 (d, 1H,  $J = 15.6$  Hz, at  $\alpha\text{-C}$  of Chalcone), 6.91 (d, 2H,  $J = 8.4$  Hz, Ar-H), 6.93-7.29 (d, 2H,  $J = 8.8$  Hz, Ar-H), 7.54 (d, 1H,  $J = 15.6$  Hz, at  $\beta\text{-C}$  of Chalcone), 8.03-8.09 (d, 2H,  $J = 8.4$  Hz, Ar-H), 8.12-8.14 (d, 2H,  $J = 8.6$  Hz, Ar-H).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ) in ppm for Dodecyloxy derivative ( $\text{C}_{12}$ ). 0.88-0.93 (t, 3H,  $-\text{CH}_3$  of  $-\text{OC}_{12}\text{H}_{25}$  group), 1.18-1.29 (m, 18H,  $2\times\text{CH}_2$  of  $-\text{OC}_{12}\text{H}_{25}$  group), 1.31-1.58 (m, 2H, Ar-O- $(\text{CH}_2)_2\text{-CH}_2$ ), 1.72-1.94 (m, 2H, Ar-O- $(\text{CH}_2)\text{-CH}_2$ ), 3.98-4.05 (t, 2H, Ar-O- $\text{CH}_2$ ), 6.88 (d, 1H,  $J = 15.6$  Hz, at  $\alpha\text{-C}$  of Chalcone), 6.95 (d, 2H,  $J = 8.4$  Hz, Ar-H), 6.97 (d, 2H,  $J = 8.8$  Hz, Ar-H), 7.27 (d, 1H,  $J = 15.6$  Hz, at  $\beta\text{-C}$  of Chalcone), 8.07 (d, 2H,  $J = 8.4$  Hz, Ar-H), 8.09 (d, 2H,  $J = 8.6$  Hz, Ar-H).

### 3. RESULTS AND DISCUSSION

4-Hydroxy Cinnamic acid and 4-n-alkoxy Cinnamic acid are nonmesomorphic. However, on condensing the two nonmesomorphic components through acid chlorides, the resultant homologous ethylene derivatives from hexyloxy to hexadecyloxy give rise to mesomorphic behavior. The methyl to pentyl homologs are nonmesomorphic. The decyloxy and dodecyloxy homologs are enantiotropically smectogenic in addition to nematogenic in character and the rest of the homologs are enantiotropic nematic only (Table 2). The transition and melting temperatures of all the homologs were determined on an optical polarizing microscope equipped with a heating stage. A plot of the transition temperatures versus number of carbon atoms present in n-alkoxy terminal chain shows the phase behavior of the series (Fig. 3). The smectic-nematic transition curve initially rises, passes through maxima and then descends as series is ascended up to the dodecyloxy homolog. The smectic mesophase formation should occur at  $109^\circ\text{C}$ . However, practically it is a solid-nematic transition temperature, which suggests that the smectic mesophase ceases to form and only the nematic mesophase forms. The nematic-isotropic transition curve descends as the series is ascended and behaves in normal manner. The solid mesomorphic or isotropic curve follows a zigzag path of rising and falling values as the series is ascended with an overall falling tendency. Thus, all the three transition curves behave in normal manner.

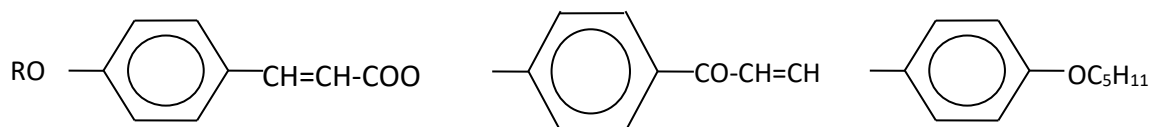
**Table 2.** Transition temperatures of series

Compound	R (n-alkyl)	Transition temperature, $^\circ\text{C}$		
		Sm	Nm	Isotropic
1	Methyl	-	-	180.00
2	Ethyl	-	-	150.00

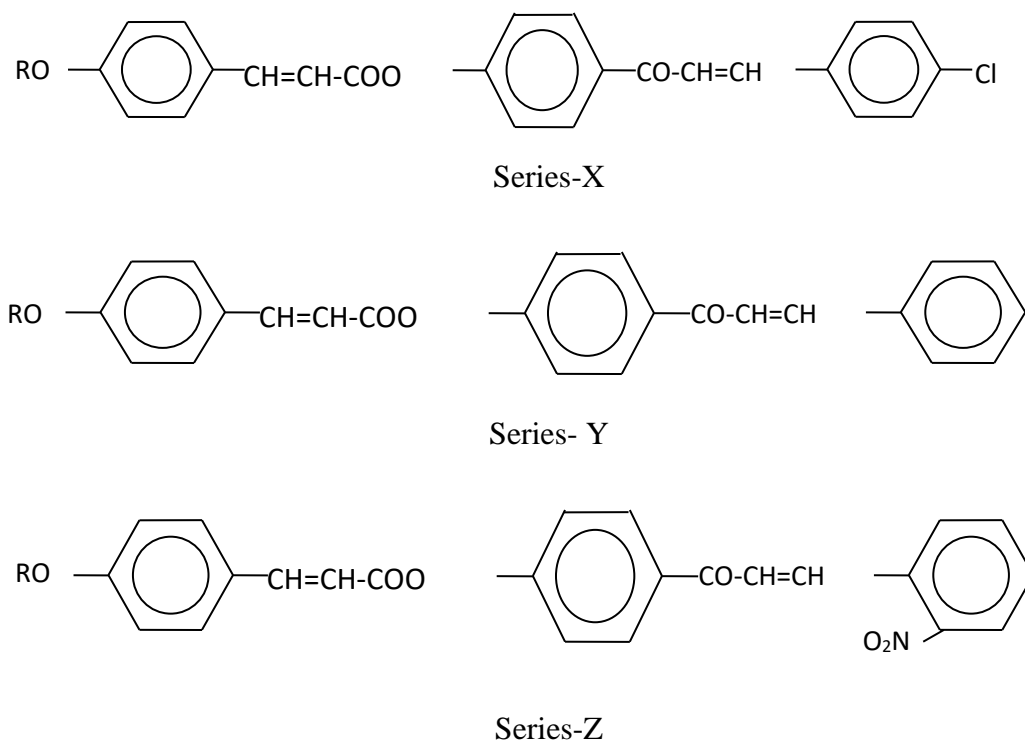
3	Propyl	-	-	158.00
4	Butyl	-	-	110.00
5	Pentyl	-	-	99.00
6	Hexyl	-	109	120.00
7	Octyl	-	119	165.00
8	Decyl	127	140	169.00
9	Dodecyl	117	132	168.00
10	Tetradecyl	-	125	162.00
11	Hexadecyl	-	135	158.00

The texture of the nematic mesophase is threaded or Schlieren and that of a smectic mesophase is of the typical A or C type. The smectic mesophase varies from a minimum of 13 °C at the decyloxy homolog to a maximum of 15 °C at the dodecyloxy homolog. The nematic mesophase length varies from a minimum of 11 °C at the hexyloxy homolog and maximum of 46 °C at the octyloxy homolog. The present series is predominantly nematogenic and partly smectogenic with a middle-ordered melting type. The smectic and nematic mesophase commence from the decyl and hexyl homolog derivatives, respectively.

The hexyl to hexadecyl homologs are mesomorphic due to their suitable magnitudes of rigidity and flexibility. The decyloxy and dodecyloxy homologs generate a lamellar packing of the molecules at a transition temperature  $t_1$ , and then adopt a statistically parallel orientational order from and beyond a higher temperature  $t_2$ . However, on further continued heating of the sample, the molecules randomly orient in all possible directions without any molecular ordering at a higher temperature  $t_3$ . Thus, the samples under microscopic examination exhibit and show a smectic mesophase between  $t_1$  and  $t_2$ , and then a nematic mesophase between  $t_2$  and  $t_3$ , and an isotropic liquid beyond temperature  $t_3$ . Mesomorphic molecules of homologs other than octyloxy to dodecyloxy exhibit only nematogenic mesomorphism. The methyl to butyl homologs of the series are unable to withstand exposed thermal vibrations due to the unsuitable magnitudes of anisotropic intermolecular forces of attraction as a consequence of unfavorable molecular rigidity and flexibility [1-7]. The odd-even effect diminishes for higher homologs, because longer n- alkyl chain from and beyond dodecyloxy homolog may coil, bend, flex or couple to lie



Series-1



**Figure 3.** Structurally similar series.

With the major is of the core [4, 8-21]. The degree of mesomorphism exhibited by the series is sufficiently high, because the series under discussion has sufficient lateral and terminal end-to-end attractions by the linear shape with two central groups bearing a conjugating double bond which strengthens rigidity of a molecule. The variation in mesomorphic property from homolog to homolog is attributed to these sequentially added methylene unit of the n-alkoxy terminal chain. The variation in mesomorphic properties is compared with other structurally similar homologous series X[11] Y[12] and Z [13] as shown in Fig. 3.

Figure 3 shows the structurally similar series 1, X, Y and Z are identical in respect of three phenyl rings bridged through to central groups-CH=CH-COO-and -CO-CH=CH- with a common left terminal end group -OR. However, the uncommon part is a right- handed terminal and groups para-C<sub>5</sub>H<sub>11</sub>, -Cl, -H, and ortho-NO<sub>2</sub>. Therefore, the molecular Aromaticity and rigidity remains identical, but the molecular flexibility varies due to polarity and induced polarizability of laterally and terminally substituted group only on third phenylring, viz.-C<sub>5</sub>H<sub>11</sub>, -NO<sub>2</sub>, -Cl, and -H. Thus, suitable magnitudes of anisotropic forces or intermolecular adhesion as a consequence of varying molecular flexibility differ accordingly. Thus, Mesomorphic properties and the degree of mesomorphism vary with changing of the terminal end groups of series 1, X, Y, and Z respectively. Thermal stability of each series and commencement of mesophase are given in Table 3.

Table 3 indicates that present series-1 and X are smectogenic in addition to nematogenic character, while series Y and Z chosen for comparison are nematogenic only without exhibition of any smectogenic character. The absence of smectogenic character in series Y and Z chosen

for comparison indicates the absence of lamellar packing of molecules in their crystal lattices. The commencement of smectogenic property takes place from the octylhomolog of series 1 and X, but it does not commence until the last homolog of the series Y and Z. This variation may be attributed to the extent of non co planarity [9] caused by the molecules concerned.

**Table 3.** Average thermal stabilities in °C

Average transition temperatures in °C				
Series	Series-1	Series-X	Series-Y	Series-Z
Smectic-nematic or smectic-isotropic	122 °C (C <sub>10</sub> -C <sub>12</sub> )	132.6 (C <sub>8</sub> -C <sub>12</sub> )	-	-
Commencement of smectic mesophase	C <sub>10</sub>	C <sub>8</sub>	-	-
Nematic-isotropic	157 °C, (C <sub>6</sub> -C <sub>16</sub> )	156.7 (C <sub>5</sub> -C <sub>16</sub> )	155.4 (C <sub>5</sub> -C <sub>16</sub> )	171.5 (C <sub>5</sub> -C <sub>16</sub> )
Commencement of nematic mesophase	C <sub>6</sub>	C <sub>5</sub>	C <sub>5</sub>	C <sub>5</sub>

The -Cl terminal end group is more polar and mono atomic, which reduces the extent of molecular non co planarity than the differing end group -OC<sub>5</sub>H<sub>11</sub>, -H or lateral group -NO<sub>2</sub>. Thus, suitable and favorable extent of molecular non co planarity facilitates lamellar packing of the molecules from the octyloxy to dodecyloxy homologs of series-1 but it does not facilitate for any homolog of series X, Y and Z. The nematic mesophase formation commences identically from the Pentyl homolog of the series X, Y and Z but from hexyl homologue of the series-1 under comparison.

This indicates that suitable magnitudes of anisotropic intermolecular forces of attraction, as emerging from molecular rigidity and flexibility, operate equally on end to end attractions, though the thermal stability of series Z is higher among those under comparison. Suitable magnitudes of anisotropic intermolecular forces of attraction facilitate a statistically parallel orientational order of the molecules which commences from the fifth homolog of series X, Y and Z to generate the nematic mesophase and sixth of series-1, Thus the variation of Mesomorphic properties for the same homolog from series to series is attributed to the unchanging terminal and/or lateral end group of each individual homologous series. The group efficiency order derived on the basis of (a) thermal stability and (b) early commencement of the mesophase is mentioned below in the conclusion.

#### 4. CONCLUSIONS

(a) Smectic group efficiency order: -Cl>-OC<sub>5</sub>H<sub>11</sub>> -NO<sub>2</sub> [ortho]

(b) Nematic group efficiency order: -NO<sub>2</sub>[ortho]>-Cl>-OC<sub>5</sub>H<sub>11</sub>>-H Smectic group efficiency order: -Cl>-OC<sub>5</sub>H<sub>11</sub>>-NO<sub>2</sub> [ortho]

- 1) Nematic group efficiency order  $-OC_5H_{11} = -Cl = -H = -NO_2[\text{ortho}]$
- 2) Variations of mesomorphic properties from homolog to homolog in the same depends upon the sequentially added methylene unit.
- 3) There fore the series is predominantly nematogenic and partly smectogenic with middle ordered melting type.
- 4) Molecular rigidity and/or flexibility dictate the mesomorphic behavior of a sub- stance.
- 5) Molecular structures that generate suitable magnitudes of anisotropic intermolecular forces of attraction as a consequence of molecular rigidity and flexibility generate mesomorphic properties in a substance.

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