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# Mesomorphism and Molecular Structure: Novel Homologous Series 4-(4'-*n*-alkoxy cinnamoyloxy)-4''-methoxy Benzyl Benzoates

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*A novel homologous series consists of 11 homologues, studied with a view to understand and establish the relations between mesomorphism and molecular structure of novel substances. All the novel members of a series except methoxy and pentyloxy homologue derivatives are liquid crystalline [LC] in nature. Mesomorphism commences from a second member of a series ethoxy, propyloxy, butyloxy, and hexyloxy to hexadecyloxy homologue derivatives are nematogenic while decyloxy, dodecyloxy, and tetradecyloxy homologue derivatives are smectogenic, in addition to nematogenic. All mesogenic homologues of a series are enantiotropic mesomorphic. Phase transition curve for Smectic-Nematic behave in normal manner without exhibition of odd-even effect, whereas nematic-isotropic transition curve adopts serpentine shape as rising and falling nature with exhibition of very minor odd-even effect. Textures of nematic phase are threaded or schlieren and that of a smectic mesophase is a focal conic fan shaped of the type smectic A or C. Analytical and spectral data supported molecular structures of homologues. The mesomorphic behaviors of present series are compared with a structurally similar other known series. Smectic and nematic thermal stabilities are 182.6 and 246.0°C, respectively. Present series is partly smectogenic and predominantly nematogenic with considerable degree of mesomorphism (maximum 100 °C) and high melting type.*

**Keywords** Enantiotropy; liquid crystals; mesomorphism; nematic; smectic

## Introduction

The ester derivatives of alkoxy benzoic and cinnamic acids are bioactive [1] molecules, which are useful for pharmaceuticals and medicinal preparations [2, 3]. Liquid crystal properties can be induced by joining 4-hydroxy phenyl benzyl benzoate (or its substituted benzyl benzoate) with an aromatic acid chloride by an appropriate synthetic method, otherwise a diether forms instead of the ester derivative. Benzyl esters of aromatic hydroxy carboxylic acids are useful as color developers for heat sensitive recording materials [4], which on linking it with *n*-alkoxy aromatic carboxylic acid chloride can yield liquid crystal bioactive materials [5, 6] containing three phenyl rings and two ester groups. Thus novel substances may exhibit LC properties [7–9] and higher potency of biological activity. The present study is restricted to synthesize novel double ester molecules and their evaluation of mesogenic properties as a novel homologous series with characterization of homologues

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which consist of three phenyl rings bonded through  $-\text{CH}=\text{CH}-\text{COO}-$  and  $-\text{COO}-$  central bridges as rigid core and two terminal groups,  $-\text{OC}_n\text{H}_{2n+1}$  and  $-\text{OCH}_3$  as left and right end groups as flexible components of the molecule respectively, whose combined effects of molecular rigidity and flexibility [10–12] operate the biometric and mesomorphic behaviors [13–15] of the substance. Hence, the present investigation is planned with a view to understand and establish the effects of molecular structure on mesomorphism phenomenon [16].

## Experimental

### Synthesis

4-Hydroxy cinnamic acid was alkylated by suitable alkylating agents ( $\text{C}_n\text{H}_{2n+1}\text{X}$ ) to form *n*-alkoxy cinnamic acids by a modified method of Dave and Vora [17] as constituent component (A) of a homologous series. Component (B) of (double) ester homologues was prepared by reacting 4-hydroxy benzoic acid and 4-methoxy benzyl alcohol by the method [18] of a European patent (m.p.  $180^\circ\text{C}$ , yield is 33%). Final (double) ester homologues were prepared by condensing components (A) individually through the acid chloride and (B) in dry cold pyridine [19]. Final products of the novel homologous series were individually decomposed filtered, washed, dried, and crystallized until they gave constant transition temperatures. The chemicals, 4-methoxy cinnamic acid, 4-hydroxy benzoic acid, 4-methoxy benzyl alcohol, dibutyltin oxide, pyridine, alkyl halides, MeOH, EtOH, thionyl chloride, HCl, and KOH were used as received, except solvents, which were dried and distilled prior to use. The synthetic route to the series is shown in scheme-1.

### Characterization

Some selected members of a present novel series were characterized by elemental analysis, infra red spectra,  $^1\text{H}$ NMR spectra and mass spectra. Micro analysis was performed by Perkin-Elmer PE 2400 CHN analyzer,  $^1\text{H}$ NMR spectra were recorded on a Bruker spectrometer using DMSO solvent. Infra red spectra were recorded on Perkin Elmer spectrum GX instrument. Transition temperatures and the textures of smectic and nematic mesophases as determined by a miscibility method, which were characterized by an optical polarizing microscope equipped with a heating stage. Thermodynamic quantity enthalpy change ( $\Delta H$ ) and entropy change ( $\Delta S = \Delta H/T$ ) are qualitatively discussed instead of DSC scan.

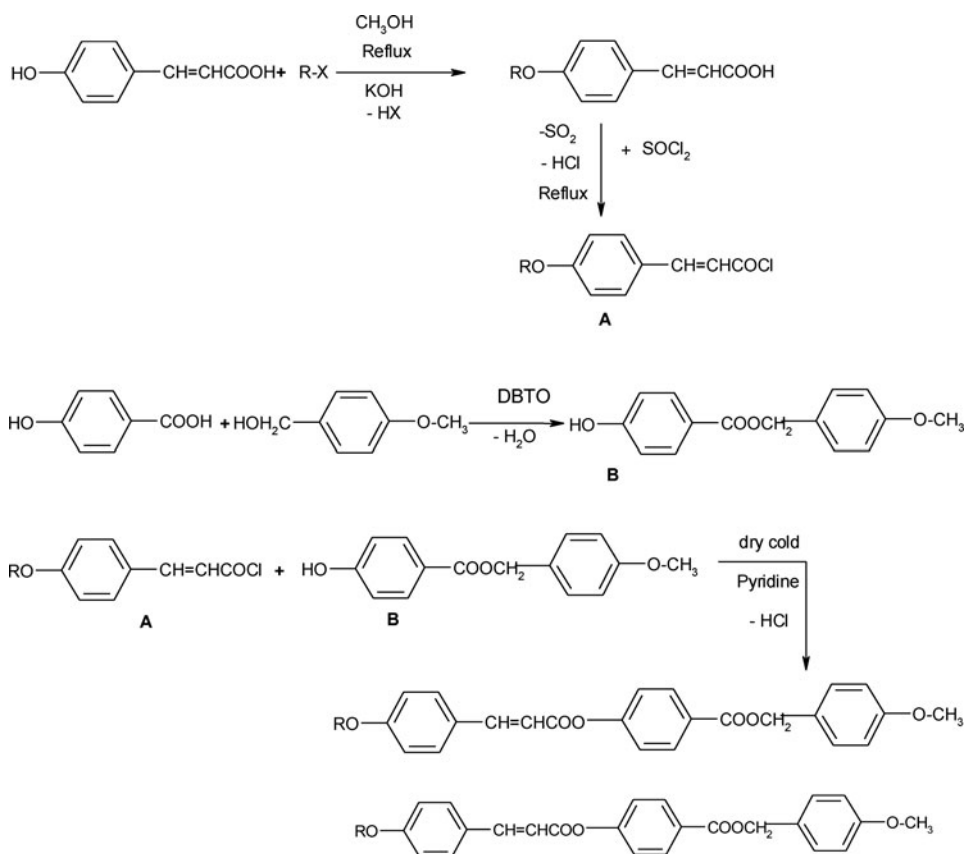
### Analytical Data

#### Spectral Data

$^1\text{H}$ NMR in ppm for the Hexyloxy Derivative: 0.83–1.66 (H of  $\text{C}_6\text{H}_{13}$ ), 2.45 ( $-\text{O}-\text{CH}_2-\text{CH}_2$  of  $-\text{OC}_6\text{H}_{13}$ ), 3.67 ( $-\text{OCH}_3$ ), 3.92 ( $-\text{OCH}_2$  of  $-\text{OC}_6\text{H}_{13}$ ), 4.98 (trans  $-\text{CH}=\text{CH}-$  group), 6.53 (p-substituted phenyl ring), and 6.83–7.97 (presence of more benzene rings).

$^1\text{H}$ NMR in ppm for the Dodecyloxy Derivative: 1.1 (H of  $\text{C}_{12}\text{H}_{25}$ ), 2.45 ( $-\text{O}-\text{CH}_2-\text{CH}_2$  of  $-\text{OC}_{12}\text{H}_{25}$ ), 3.64 ( $-\text{O}-\text{CH}_2$  of  $-\text{OC}_{12}\text{H}_{25}$ ), 3.41 ( $-\text{OCH}_3$ ), 3.92 ( $-\text{COO}-\text{CH}_2-\text{C}_6\text{H}_4$ ), 4.98 (trans  $-\text{CH}=\text{CH}-$  group), 6.2–6.44 (p-substituted phenyl ring), and 6.83–7.96 (presence of more benzene rings).

IR in  $\text{cm}^{-1}$  for Octyloxy Derivative: 825 para-substituted phenyl ring, 1123 ether linkage of ( $-\text{C}_8\text{H}_{17}\text{O}-\text{C}_6\text{H}_4$ ), 1032, 1244, 1685 ( $-\text{COO}-$  ester group), 960 (trans  $-\text{CH}=\text{CH}-$  group), and 3447 (broad signal indicates  $-\text{OH}$  group).



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

Series: 4-[4'-*n*-Alkoxy cinnamoyloxy]-4'-methoxy benzyl benzoates,  
where  $R = \text{C}_n\text{H}_{2n+1}$ ;  $n = 1, 2, 3, 4, 5, 6, 8, 10, 12, 14, 16$ .

*IR in  $\text{cm}^{-1}$  for Tetradecyloxy Derivative:* 721 polymethylene of  $\text{C}_{14}\text{H}_{29}$ , 821 para-substituted phenyl ring, 1018 ether linkage of  $(-\text{C}_{14}\text{H}_{29}\text{O}-\text{C}_6\text{H}_4)$ , 1173, 1217, 1650 ( $-\text{COO}-$ ester group), and 980 (trans  $-\text{CH}=\text{CH}-$  group).

### Mass Spectra

*Mass Spectra for Decyloxy Derivative:* Molecular formula  $\text{C}_{34}\text{H}_{40}\text{O}_6$  calculated molecular weight 544. Practical mass observed by mass spectroscopy 544.

### Texture of Nematic and Smectic Phase by Miscibility Method

Butyloxy derivative  $\longrightarrow$  Threaded nematic  
 Octyloxy derivative  $\longrightarrow$  Schlieren nematic  
 Dodecyloxy derivative  $\longrightarrow$  Smectic A  
 Tetradecyloxy derivative  $\longrightarrow$  Smectic C

**Table 1.** Elemental analysis for methyloxy, ethyloxy, and propyloxy derivatives

S. no.	Molecular formula	Elements % found (% calculated)	
		C	H
1	C <sub>25</sub> H <sub>22</sub> O <sub>6</sub>	71.35 (71.77)	5.10 (5.26)
2	C <sub>26</sub> H <sub>24</sub> O <sub>6</sub>	72.05 (72.22)	5.37 (5.55)
3	C <sub>27</sub> H <sub>26</sub> O <sub>6</sub>	74.54 (72.64)	5.65 (5.82)

## Results and Discussion

The constituent components forming homologues of the novel homologous series of *cis*-4-*n*-alkoxy cinnamic acid and 4-hydroxy-4'-methoxy benzyl benzoate are nonliquid crystal (NLC) components. However, on condensing two NLC components, yielded mesomorphism from the second member of the series. All the members of the novel series except the first and the fifth members, exhibit either only nematogenic or smectogenic with the nematogenic mesophase in enantiotropic manner. The decyl, dodecyl, and tetradecyl homologues are smectogenic in addition to nematogenic and the rest of the mesogenic homologues including smectogenic homologues are nematogenic. Thus, the present series is predominantly nematogenic and partly smectogenic. Transition temperatures of the series (Table 2) as determined by an optical polarizing microscopy are plotted versus the number of carbon atoms present in *n*-alkyl chain of left *n*-alkoxy terminal end group. Transition curves viz. solid-isotropic/mesomorphic, smectic-nematic and nematic-isotropic are obtained by joining like or related points, which formed a phase diagram (Fig. 1) of the series showing the phase behavior of the series. The solid-isotropic/mesomorphic transition curve follows a zigzag path of rising and falling values in the usual established manner. The smectic-Nematic transition curve initially rises and then falls after passing through maxima, without the exhibition of an odd-even effect and behaved in normal manner. The nematic-isotropic,

**Table 2.** Transition temperatures of series in °C

Compound no.	<i>n</i> -alkyl group (—C <sub>n</sub> H <sub>2n+1</sub> ) (n)	Transition temperature in °C		
		<i>Sm</i>	<i>Nm</i>	Isotropic
1	1	—	—	198.0
2	2	—	195.0	243.0
3	3	—	209.0	228.0
4	4	—	208.0	226.0
5	5	—	—	202.0
6	6	—	154.0	254.0
7	8	—	188.0	266.0
8	10	186.0	215.0	242.0
9	12	182.0	222.0	262.0
10	14	180.0	188.0	217.0
11	16	—	182.0	276.0

*Sm*: Smectic *Nm*: Nematic.

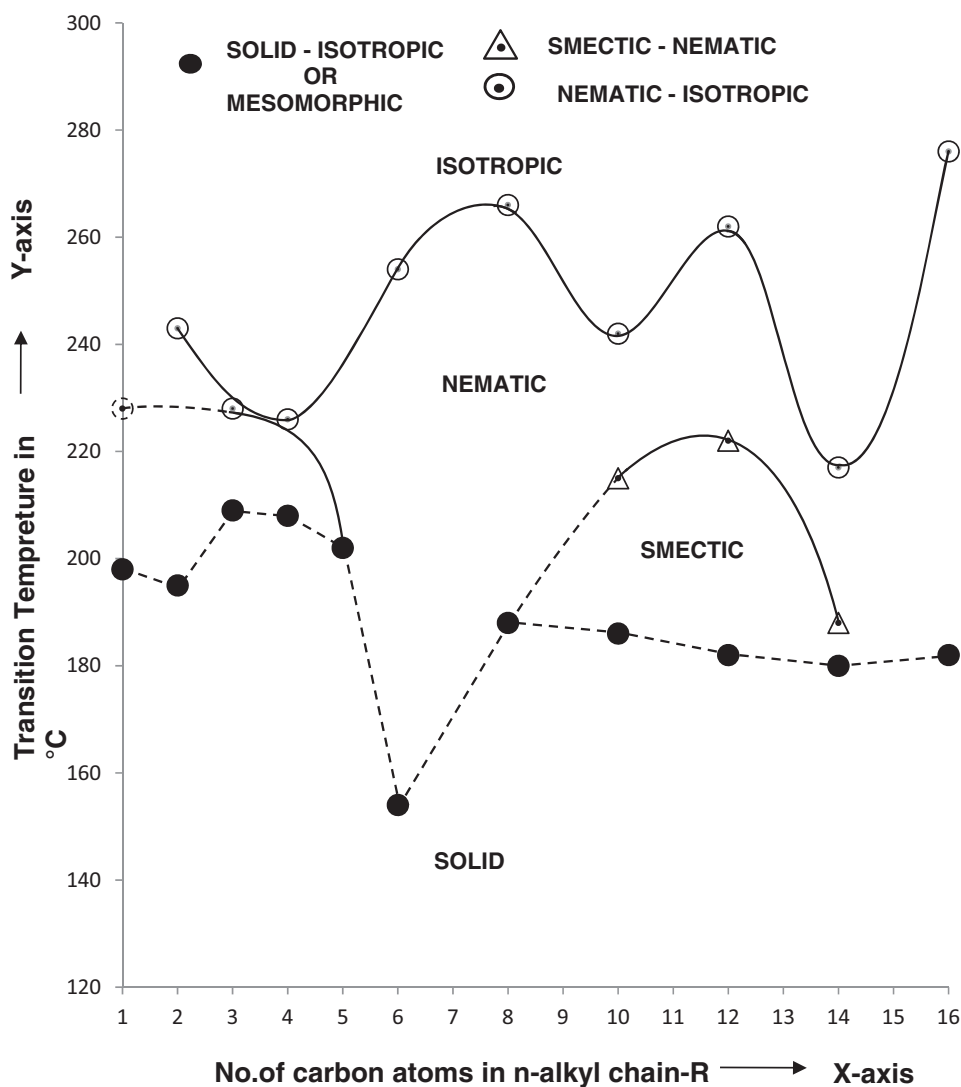
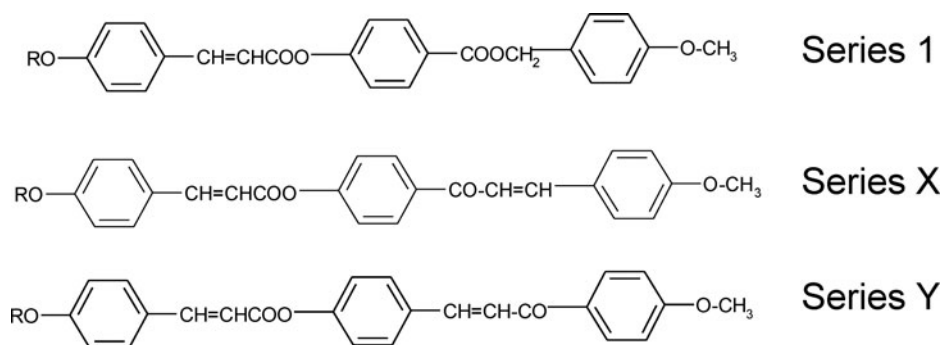


Figure 1. Phase behavior of series.

transition curve deviates from normal behavior and adopted a path of rising and falling values as the series is ascended for even members of the series with (i) exhibition of a wave shaped appearance, and (ii) odd-even effect of about 5°C. The nematic-isotropic transition curves for the odd and the even members of the series merge into each other at the butyloxy homologue and then curve for odd members from the pentyloxy homologue and extrapolated [20–22] to first member of the series to magnify the odd-even effect for nematic-isotropic transition curve. The textures of the nematic mesophase are threaded or Schlieren as determined by a miscibility method, whereas, that of the smectic phase are judged directly by observing mesophase through a polarizing microscope. The smectic and nematic thermal stabilities of the series are 182.6 and 246°C, whose mesophase length varies between 18 and 100°C at the fourth and sixth homologues, respectively. The isotropic

temperatures of the mesomorphic homologues vary between 276 and 217°C. Thus, novel homologous series is a high melting type with sufficient mesophase length. On condensing two nonmesomorphic molecules, the molecular length, molecular polarity, and polarizability and hence suitable magnitudes of anisotropic forces of intermolecular adhesion as a consequence of favorable molecular rigidity and flexibility fitted in such a manner to facilitate mesomorphism in the majority of members (nine) of the series, either only as nematogenic or along with smectogenic behavior. Nonmesomorphic behavior of the odd members, viz., first and fifth member of a series is observed due to the *n*-alkyl chain of the left *n*-alkoxy terminal end group, which hinders the suitable magnitudes of anisotropic forces of intermolecular attractions to induce mesomorphism, but induced high crystallizing tendency. Hence, nonmesomorphic homologues are passing directly into isotropic liquid from solid crystalline state, avoiding mesophase formation. Exhibition of mesomorphic behavior of a substance is attributed to the respective sample substance against exposed thermal vibrations arising from suitably favorable magnitudes of molecular rigidity and flexibility, which maintain molecular disalignment at an angle of less than ninety degree for the definite range of temperature, to float on the surface with a definite two-dimensional ordered arrangement. The molecules of decyl, dodecyl, and tetradecyl homologue derivatives possess lamellar packing of molecules in their crystal lattices and maintained sliding layered arrangement of molecules in floating condition between two temperatures with exhibition of enantiotropically smectogenic character. Then, the molecules of the same homologues floated with the statistically parallel orientational order on the surface for a range of temperature facilitating nematogenic character. Enantiotropic homologues display a mesogenic phase reversibly at a temperature at which the mesophase disappears on cooling the melt. The rest of the mesomorphic homologues show only nematogenic character by floating on the surface in a manner as described above. Missing of odd-even effect for smectic-nematic transition curve is due to the late commencement of smectic mesophase, and unfavorable extent of noncoplanarity caused by the molecules. The odd-even effect for the nematic-isotropic transition curve is observed from the third homologue and merges at the fourth homologue. Then on extending it to an odd member, i.e., to fifth member, the curve separated from an even membered curve and coincided with the solid-isotropic point of the pentyloxy homologue. Such behavior of odd members indicates that the solid-nematic and nematic-isotropic point of temperatures (202°C) match with each other at the same point. The nematic-isotropic transition curve is extrapolated [23] to the first member of the series for the purpose of magnification of the odd-even effect keeping in mind, the trend of the nematic-isotropic transition curve for odd members of the series. The smectic-nematic transition curve extrapolated to the left toward the octyloxy homologue, which merges into the solid-nematic transition point of temperature (188°C) indicating that a smectic mesophase is absent prior to nematic phase. Thus, the mesomorphic properties vary from homologue to homologue for the presently investigated novel series due to sequential addition of methylene units. The nematogenic and smectogenic phase lengths are vary from 18.0 to 100.0°C and 8.0 to 40.0°C, respectively. Thus, the present novel series is predominantly nematogenic and partly smectogenic whose degree of mesomorphism is relatively wider and of a high melting type. Mesomorphic properties of present novel series-1 are compared with structurally similar known isomeric series X [24] and Y [25] chosen for comparison as shown above in Fig. 2.

Homologous novel series-1 under discussion and isomeric homologous series X and Y selected for comparison are structurally similar with respect to two terminal end groups of -OR and -OCH<sub>3</sub> inducing similar molecular flexibility. However, they differ with respect to molecular rigidity due to the difference arising from the central linking group of



**Figure 2.** Structurally similar homologous series.

$-\text{COO}-\text{CH}_2-$ ,  $-\text{CO}-\text{CH}=\text{CH}-$ , and  $-\text{CH}=\text{CH}-\text{CO}-$  for series-1, X and Y, respectively. Three phenyl rings and one of the central group  $-\text{CH}=\text{CH}-\text{COO}-$  contribute to molecular rigidity similarly, but the total molecular rigidity differs due to difference of the other linking group. Therefore, the combined effect of molecular rigidity and flexibility responsible for inducing mesomorphic behavior and the degree of mesomorphism vary from series to series, under comparison as shown in Fig. 2. Table-3 shows the thermal stabilities for smectic and nematic phases, group efficiency order based on thermal stability and early commencement of a mesophase.

The data in Table 3 shows the following.

- Homologous series-1 and X exhibit smectogenic character while homologous series Y does not show smectogenic character.
- All the homologous series 1, X and Y under comparison are enantiotropically nematogenic.
- Average thermal stabilities for smectic and nematic mesophases decreases from series 1 to X to Y.
- The smectic and nematic mesophases for the presently investigated novel homologous series-1 commences from the decyloxy and ethoxy homologue derivatives, respectively.

**Table 3.** Average thermal stability in  $^{\circ}\text{C}$

Series→	Series 1	Series X	Series Y
Smectic-nematic Or Smectic-isotropic	182.6 ( $\text{C}_{10} - \text{C}_{14}$ )	159.7 ( $\text{C}_5 - \text{C}_8$ )	—
Commencement of Smectic phase	$\text{C}_{10}$	$\text{C}_5$	
Nematic-isotropic Commencement of nematic phase	242.0 ( $\text{C}_2 - \text{C}_{16}$ ) $\text{C}_2$	174.1 ( $\text{C}_5 - \text{C}_{14}$ ) $\text{C}_5$	166.6 ( $\text{C}_5 - \text{C}_{14}$ ) $\text{C}_5$



- The smectic and nematic mesophase formation commences from the pentyloxy homologue of series X and the nematic mesophase commences from the pentyloxy homologue of a series Y but the smectogenic mesophase formation does not commence until the last hexadecyloxy homologue of series Y.

Homologous series 1, X and Y are linear lath like, with differing overall magnitudes of molecular rigidity and flexibility. Therefore, suitable magnitudes of anisotropic forces of intermolecular attractions due to varying molecular length, double bond conjugation ( $-\text{CH}=\text{CH}-$ ) [26] molecular polarity and polarizability, position of  $-\text{CO}-$  group, lateral and terminal attractions, vary from series to series. Moreover, the presence (series 1 and X) or absence (series-Y) of lamellar packing of molecules in their crystal lattices and the extent of noncoplanarity of the molecules depending upon magnitudes of intermolecular adhesion vary from series to series, affecting the mesophase formation and stabilization as well as degree of mesomorphism of the series under discussion. The variations in mesomorphic properties for the **same homologue** form series to series are attributed to the differing part of the molecules. The variations in mesomorphic properties from homologue to homologue in the **same series** is attributed to the sequentially added methylene units, keeping rest of the molecular structure unchanged.

## Conclusions

- The group efficiency order derived for smectic and nematic on the basis of (i) thermal stability and (ii) the early commencement of mesophase are as under.
  - (i) Smectic:  $-\text{COOCH}_2- > -\text{CO}-\text{CH}=\text{CH}- > -\text{CH}=\text{CH}-\text{CO}-$   
 Nematic:  $-\text{COOCH}_2- > -\text{CO}-\text{CH}=\text{CH}- > -\text{CH}=\text{CH}-\text{CO}-$
  - (ii) Smectic:  $-\text{CO}-\text{CH}=\text{CH}- > -\text{COO}-\text{CH}_2- > -\text{CH}=\text{CH}-\text{CO}-$   
 Nematic:  $-\text{COO}-\text{CH}_2- > -\text{CO}-\text{CH}=\text{CH}- > -\text{CH}=\text{CH}-\text{CO}-$
- Variations in mesomorphic properties from homologue to homologue in the same series are due to the sequentially added methylene units.
- Variations in mesomorphic properties for the same homologue from series to series is due to the changing molecular structure, such as the terminal end group or central group or the number of phenyl rings, or the position of a group or changing polarity or molecular linearity or nonlinearity.
- Suitable magnitudes of intermolecular attractions are a consequence of favorable molecular rigidity and flexibility.
- Suitable magnitudes of molecular rigidity and flexibility are the main source of the resulting mesomorphism phenomenon.
- Molecular rigidity and flexibility are very sensitive and susceptible to changes in molecular structure.

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