CHAPTER-2
EXPERIMENTAL

The following new homologous series of liquid crystal have been synthesized. All the homologous series under present investigation are mesomorphic in character.

Eight homologous series of new substance were synthesized.

The following homologous series have been newly synthesizes.

**Series-1:** $\alpha$-$4'$-[4'-n-Alkoxy- Cinnamoyloxy] Benzoyl-$\beta$-3” Nitro phenyl Ethylenes.

**Series-2:** $\alpha$-$4'$-[4'-n-Alkoxy Cinnamoyloxy] benzoyl $\beta$-3”,4”-dimethoxy Phenyl Ethylenes.

**Series-3:** $\alpha$-$4'$(-$4'$-n-Alkoxy Cinnamoyloxy) phenyl $\beta$-4”-Nitrobenzoyl Ethylenes.

**Series-4:** $\alpha$-$3'$-[4'-n-Alkoxy Cinnamoyloxy] phenyl-$\beta$-4”’- Nitrobenzoyl Ethylenes.

**Series-5:** $\alpha$-$4'$(-$4'$-n-Alkoxybenzoyloxy] Benzoyl-$\beta$-3” Nitro phenyl Ethylenes.
Series-6: $\alpha$-4-[4’-n-Alkoxy benzoyloxy] benzoyl $\beta$-3”,4”-dimethoxy Phenyl Ethylenes.

Series-7: Series: $\alpha$ – 4-[4’-n-Alkoxy benzoyloxy] phenyl- $\beta$-4”-nitro benzoyl ethylenes.

Series-8: $\alpha$ – 3-[4’-n-Alkoxy benzoyloxy] phenyl- $\beta$-4”-nitro benzoyl ethylenes.

The above homologous series of new mesogens have been synthesized by adopting following different steps.

2.1 Preparation of Compounds :

2.1.1. $\alpha$-4-[4’-n-Alkoxy- Cinnamoyloxy] Benzoyl-$\beta$-3” Nitro phenyl Ethylenes
2.1.1(a) Synthesis of n-alkyl halides:

Corresponding purified alcohols were used to synthesis of n-alkyl halides by standard methods[88] and pure products were obtained, from ethyl to hexyl halide. Transition points are satisfactorily agreed with the reported literature. Higher alkyl halides viz heptyl, octyl, decyl, dodecyl, tetradecyl, hexadecyl, were directly purchased from market and used as received.

2.1.1(b) Synthesis of p-hydroxy cinnamic acid:

It was prepared by dissolving p-hydroxy benzaldehyde (0.15 mole) and malonic acid (0.375 mole) in pyridine (50.0 ml) to which few drops of piperidine added and then warmed for a rapid evolution of CO\(_2\) take place. The reaction completed by boiling the solution for five minutes. It was cooled and poured into excess of water containing enough HCl to remove excess of pyridine. The p-hydroxy cinnamic acid was separated and filtered off, washed with water and recrystallized from alcohol several times as white shining crystals. The fine crystals obtained were melted sharply at their reported melting point 214\(^\circ\)C yield is 69.9%.

2.1.1(c) Synthesis of Cis p-n-alkoxy cinnamic acids:

Number of methods [89] are known for alkylation of p-hydroxy benzoic acid and p-hydroxy cinnamic acid. p-Hydroxy cinnamic acid was alkylated by the treatment of corresponding alkyl halide with p-Hydroxy acid by the established method of Dave and Vora [89].

p-Hydroxy cinnamic acid (0.1 mole), corresponding alkyl halide (0.12 mole) and potassium hydroxide (0.25 mole) were dissolved in 100 ml CH\(_3\)OH and reaction mixture was refluxed for three to four hours, 10% aqueous potassium hydroxide solution (20 ml) was added and refluxing continued for further 2 hours to hydrolyse any ester formed. The solution was cooled and acidified with 1:1 hydrochloric acid to precipitate the corresponding alkoxy cinnamic acids. Time period for refluxing of reaction mixture was extended with increasing alkyl chain length. Cinnamic acids were crystallized from CH\(_3\)OH or CH\(_3\)COOH till constant melting points were obtained.

2.1.1(d) Synthesis of p-n-alkoxy cinnamoyl chlorides:

They were prepared by reacting the corresponding p-n-alkoxy cinnamic acids with excess of freshly distilled SOCl\(_2\) and heating on a water bath till the evolution of HCl and SO\(_2\) ceased. Excess of SOCl\(_2\) was distilled off and the acid chloride left behind preserved in flask in moisture free atmosphere and directly treated for next reaction without further purification.

2.1.1(e) α-p-Hydroxy benzoyl β-m'-Nitro phenyl ethylene:
m-nitro benzaldehyde (0.1mole) and p-hydroxy acetophenone (0.1mole) were dissolved in minimum amount of ethanol 50% KOH solution was added to the above solution[90]. The flask was heated at 50°C for twenty hours or room temperature overnight. The solution was acidified by cold 1:1 HCl. Crystalline solid product with light yellow colour was filtered and washed with water. The p-hydroxy benzoyl β-m'-Nitro phenyl ethylene was recrystallized from ethanol M.P. is170°C and yield is 60%.

2.1.1 Final product series-1

α-p-hydroxy benzoyl β-m'-nitro phenyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy cinnamoyl chloride (0.015 mole)[90,91]. The reaction mixture was warmed on water bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 HCl. The precipitates were filtered, washed with H2O, 10% aqueous NaHCO3 solution and again with H2O. The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 69.0%.

The transition temperatures, elemental analysis and data of spectral are recorded in table-2, 3 and 4(a) and 4(b) respectively.

2.1.2 α-4-[4'-n-Alkoxy Cinnamoyloxy] benzoyl β-3”,4”-dimethoxy Phenyl Ethylenes

2.1.2 (a) n-alkyl halides, p-hydroxy cinnamic acid, p-n-alkoxy cinnamic acids and p-n-alkoxy cinnamoyl chlorides were synthesized as described in 2.1.1 (a), 2.1.1 (b) 2.1.1 (c) and 2.1.1 (d) respectively.

2.1.2 (b) α-p-Hydroxy benzoyl β-3’,4’-dimethoxy phenyl ethylene.

3,4-dimethoxy benzaldehyde (0.1mole) and p-hydroxy acetophenone (0.1mole) were dissolved in minimum amount of ethanol 50% potassium hydroxide solution was added to the above solution. The flask was heated at 50°C for twenty hours or room temperature overnight. The solution was acidified by cold 1:1 hydrochloric acid. Crystalline solid product with light yellow colour was filtered and washed with water. The α-4-Hydroxy benzoyl β-3’,4’-dimethoxy phenyl ethylene was recrystallized from ethanol M.P. is 185°C and yield was 65%.

2.1.2 Final product series-2

α-p-Hydroxy benzoyl β-3’,4’-dimethoxy phenyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy cinnamoyl chloride (0.015 mole)[90]. The reaction mixture was warmed on water bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 hydrochloric acid. The
precipitates were filtered, washed with water, 10% aqueous sodium bicarbonate solution and
again with water. The solid esters were crystallized from ethanol 80% and 20% benzene. The
yield was 65.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-6,7, 8(a)
and 8(b) respectively.

2.1.3 α-4(-4'-n-Alkoxy Cinnamoyloxy) phenyl β-4”-Nitrobenzoyl Ethylenes.

2.1.3(a) n-alkyl halides, p-hydroxy cinnamic acid, p-n-alkoxy cinnamic acids and p-n-
alkoxy cinnamoyl chlorides were synthesized as described in 2.1.1 (a), 2.1.1 (b) 2.1.1 (c) and
2.1.1 (d) respectively.

2.1.3 (b) α-4-hydroxy phenyl β-4’-nitro benzoyl ethylene:

p-hydroxy benzaldehyde (0.1mole) and p-nitro acetophenone (0.1mole) were dissolved in
minimum amount of ethanol 50% KOH solution was added to the above solution[90,92]. The
flask was heated at 50°C for twenty hours or room temperature overnight. The solution was
acidified by cold 1:1 HCl. Crystalline solid product with light yellow colour was filtered and
washed with water. The α-4-hydroxy phenyl β-4’-nitro benzoyl ethylene was recrystallized from
ethanol M.P. is 174°C and yield is 69%.

2.1.3 Final product series-3

α-4-hydroxy phenyl β-4’-nitro benzoyl ethylene (0.01 mole) was dissolved in dry pyridine
(10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy
cinnamoyl chloride (0.015 mole) [93]. The reaction mixture was warmed on H₂O bath for about
an hour and allowed to stand overnight. It was decomposed in cold 1:1 HCl. The precipitates
were filtered, washed with water, 10% aqueous NaHCO₃ solution and again with H₂O. The solid
esters were crystallized from ethanol 80% and 20% benzene. The yield was 65.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-10,11,
12(a) and 12(b) respectively.

2.1.4 α-3-[4’-n-Alkoxy Cinnamoyloxy] phenyl-β-4’’- Nitrobenzoyl Ethylenes.

2.1.4 (a) n-alkyl halides, p-hydroxy cinnamic acid, p-n-alkoxy cinnamic acids and p-n-
alkoxy cinnamoyl chlorides were synthesized as described in 2.1.1 (a), 2.1.1 (b) 2.1.1 (c) and
2.1.1 (d) respectively.
2.1.4 (b) \( \alpha \)-3-Hydroxy phenyl \( \beta \)-4’-nitro benzoyl ethylene:

m-hydroxy benzaldehyde (0.1mole) and p-nitro acetophenone (0.1mole) were dissolved in minimum amount of ethanol 50% potassium hydroxide solution was added to the above solution. The flask was heated at 50\(^\circ\)C for twenty hours or room temperature overnight. The solution was acidified by cold 1:1 HCl [90,92]. Crystalline solid product with light yellow colour was filtered and washed with \( \text{H}_2\text{O} \). The \( \alpha \)-3-Hydroxy phenyl \( \beta \)-,4’-nitro benzoyl ethylene was recrystallized from ethanol M.P. is 177\(^\circ\)C and yield is 69%.

2.1.4 Final product:
\( \alpha \)-3-hydroxy phenyl \( \beta \)-4’-nitro benzoyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy cinnamoyl chloride (0.015 mole) [93]. The reaction mixture was warmed on \( \text{H}_2\text{O} \)bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 HCl. The precipitates were filtered, washed with water, 10% aqueous \( \text{NaHCO}_3 \) solution and again with \( \text{H}_2\text{O} \). The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 67.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-14, 15, 16(a) and 16(b) respectively.

2.1.5 \( \alpha \)-4-[4’- n-Alkoxybenzoyloxy] Benzoyl-\( \beta \)-3” Nitro phenyl Ethylenes.

2.1.5 (a) Synthesis of n-alkyl halides:

Corresponding purified alcohols were used to synthesis of n-alkyl halides by standard methods [89] and pure products were obtained. Transition points are satisfactorily agreed with the reported literature [94].

2.1.5 (b) Synthesis of p-n-alkoxy benzoic acids:

Number of methods [95, 96] are known for alkylation of p-hydroxy benzoic acid. One of them is that which has been described by Gray and Jone [95] and Dave and Vora [96]. p-Hydroxy benzoic acid (0.1 mole), corresponding alkyl halide (0.12 mole) and potassium
hydroxide (0.25 mole) were dissolved in 100 ml CH₃OH and reaction mixture was refluxed for three to four hours, 10% aqueous potassium hydroxide solution (20 ml) was added and refluxing continued for further 2 hours to hydrolyse any ester formed. The solution was cooled and acidified with 1:1 hydrochloric acid to precipitate the corresponding alkoxy benzoic acids. Time period for refluxing reaction mixture was extended with increasing alkyl chain length. Benzoic acids were crystallized from ethanol or CH₃COOH till constant melting points were obtained. The transition temperatures are in agreement with the reported literature [97]. These are listed in Table-(1).

2.1.5 (c) Synthesis of p-n-alkoxy benzoyl chlorides:

They were prepared by reacting the corresponding p-n-alkoxy benzoic acids with excess of freshly distilled SOCl₂ and warming on a H₂O bath till the evolution of HCl gas stopped. Excess of SOCl₂ was distilled off under reduced pressure using water pump and the acid chloride left behind was directly treated for next reaction without further purification.

2.1.5 (d) α-4-hydroxy benzoyl β-3'-nitro phenyl ethylene was synthesized as described in 2.1.1(e).

2.1.5 Final product

α-4-Hydroxy benzoyl β-3'-nitro phenyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy benzoyl chloride (0.015 mole)[90,91]. The reaction mixture was warmed on H₂Obath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 HCl. The precipitates were filtered, washed with H₂O, 10% aqueous NaHCO₃ solution and again with H₂O. The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 72.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-18,19, 20(a) and 20(b) respectively.

2.1.6 α-4-[4’-n-Alkoxy benzoyloxy] benzoyl β-3”,4”-dimethoxy Phenyl Ethylenes.

2.1.6 (a) n-alkyl halides, p-n-alkoxy benzoic acids and p-n-alkoxy benzoyl chlorides were synthesized as described in 2.1.5 (a), 2.1.5 (b) and 2.1.5 (c) respectively.

2.1.6 (b) α-p-Hydroxy benzoyl β-3’,4’-dimethoxy phenyl ethylene was synthesized as described in 2.1.2(b).

2.1.6 Final product
α-4-Hydroxy benzoyl β-3′,4′-dimethoxy phenyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy benzoyl chloride (0.015 mole). The reaction mixture was warmed on water bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 hydrochloric acid. The precipitates were filtered, washed with water, 10% aqueous sodium bicarbonate solution and again with water. The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 69.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-22,23,24(a) and 24(b) respectively.

2.1.7 α – 4-[4’-n-Alkoxy benzoyloxy] phenyl- β-4’’-nitro benzoyl ethylenes.

2.1.7 (a) n-alkyl halides, p-n-alkoxy benzoic acids and p-n-alkoxy benzoyl chlorides were synthesized as described in 2.1.5 (a), 2.1.5 (b) and 2.1.5 (c) respectively.

2.1.7 (b) α-4-hydroxy phenyl β-4’-nitro benzoyl ethylene was synthesized as described in 2.1.3 (b).

2.1.7 Final product

α-4-hydroxy phenyl β-4’-nitro benzoyl ethylene (0.01 mole) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy benzoyl chloride (0.015 mole). The reaction mixture was warmed on water bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 hydrochloric acid [90,91]. The precipitates were filtered, washed with water, 10% aqueous sodium bicarbonate solution and again with water. The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 70.5%.

The transition temperatures, elemental analysis and spectral data are recorded in table-26,27,28(a) and 28(b) respectively.

2.1.8 α – 3-[4’-n-Alkoxy benzoyloxy] phenyl- β-4’’-nitro benzoyl ethylenes.

2.1.8 (a) n-alkyl halides, p-n-alkoxy benzoic acids and p-n-alkoxy benzoyl chlorides were synthesized as described in 3.5.1 (a), 3.5.1 (b) and 3.5.1 (c) respectively.

2.1.8 (b) α-3-Hydroxy phenyl β-4’-nitro benzoyl ethylene was synthesized as described in 2.1.4(b).

2.1.8 Final product
α-3-hydroxy phenyl β-4’-nitro benzoylethylene (0.01 mol) was dissolved in dry pyridine (10 ml) and was added dropwise with constant stirring to the corresponding p-n-alkoxy benzoyle chloride (0.015 mol). The reaction mixture was warmed on water bath for about an hour and allowed to stand overnight. It was decomposed in cold 1:1 hydrochloric acid[90,91]. The precipitates were filtered, washed with water, 10% aqueous sodium bicarbonate solution and again with water. The solid esters were crystallized from ethanol 80% and 20% benzene. The yield was 72.0%.

The transition temperatures, elemental analysis and spectral data are recorded in table-30,31,32(a) and 32(b) respectively

2.2 Scheme of synthesis for series-1 to 8

Series-1
Series-2

Where \( R = \text{C}_n\text{H}_{2n+1} \quad n=1,2,3,4,5,6,8,10,12,14,16. \)

Series-3

Where \( R = \text{C}_n\text{H}_{2n+1} \quad n=1,2,3,4,5,6,7,8,10,12,14,16. \)
Where R = C_{n}H_{2n+1} \quad n=1,2,3,4,5,6,8,10,12,14,16.

Series-4

Where R = C_{n}H_{2n+1} \quad n=1,2,3,4,5,6,8,10,12,14,16.

Series-5
(1) p-Hydroxy benzoic acid

R-X → KOH → Reflux → MeOH → RO-\(-\)COOH

Alkoxo benzoic acid

(2) p-Hydroxy acetophenone + \(\text{m-Nitro benzaldehyde}\)

\[ \text{Alcohol} \downarrow 20 \text{ hours} \rightarrow 60^\circ \text{C} \]

HO-\(-\)CO-CH=CH-\(-\)NO₂

(3) (A) + (B) → Final Product

\[ \text{Dry pyridine, Cold} \rightarrow 1:1 \text{ HCl} \]

Where \( R = \text{C}_n\text{H}_{2n+1} \)  \( n=1,2,3,4,5,6,8,10,12,14,16. \)

Series-6

(1) p-Hydroxy benzoic acid

R-X → KOH → Reflux → MeOH → RO-\(-\)COOH

Alkoxo benzoic acid

(2) p-Hydroxy acetophenone + \(\text{3,4-Dimethoxy benzaldehyde}\)

\[ \text{Alcohol} \downarrow 20 \text{ hours} \rightarrow 50^\circ \text{C} \]

HO-\(-\)CO-CH=CH-\(-\)NO₂

(3) (A) + (B) → Final Product

\[ \text{Dry pyridine, Cold} \rightarrow 1:1 \text{ HCl} \]

Where \( R = \text{C}_n\text{H}_{2n+1} \)  \( n=1,2,3,4,5,6,7,8,10,12,14,16. \)

Series-7
2.3 Study of the Homologous series

Final Product

Where \( R = C_nH_{2n+1} \quad n = 1, 2, 3, 4, 5, 6, 8, 10, 12, 14, 16. \)
The study of the homologous series is carried out by following two methods.

(i) Indigenous optical method and
(ii) Hot stage polarizing microscopy method.

Generally, we know that the substance melts to an isotropic liquid at its melting point. The melting point can usually be determined by commonly used method. However, a mesomorphic substance first melts to an anisotropic liquid at a particular temperature, and this anisotropic liquid transforms to a true isotropic liquid at a definite higher temperature, the point at which this change takes place is usually called the transition point or transition temperature. It is difficult to determine accurately the melting point and the transition point of a liquid crystalline substances by the general melting point method. The mesomorphic liquid being birefringent, both its melting point and transition point can very conveniently be determined by the use of Polaroid sheets. This is known as an optical method. The melting point and transition point of pure mesogens or non-mesogens as well as two component systems can be investigated by optical method.

(i) Indigenous optical method:

The apparatus used for this method is diagrammatically represented in figure-. The capillary tube C is made by drawing out an ordinary glass tubing to form a capillary of about 2 mm diameter. In this capillary tube, a small portion of the finely powdered sample (mixture or pure substance) is taken and a thin glass stirrer S is provided. This capillary tube is immersed in a liquid bath consisting of two litre beaker capacity containing dibutyl pthalate.

The liquid in the bath is continuously and vigorously stirred by means of an electrically run mechanical stirrer M. The beaker is lagged by wrapping asbestos paper round it in order to minimize the loss of heat through radiation. The asbestos covering is provided with two windows 1½” square opposite one other. A standard thermometer T, which is previously calibrated, is suspended in the liquid bath. The capillary tube C containing the pure compound or the mixture under investigation is held in the bath by the side of the thermometer in such a way that it can be seen through the square windows in the asbestos lagging.

An electrical lamp L functions as the source of light. The beam of light passes through a ground glass screen G illuminating the substances in the capillary tube C which is observed by means of a magnifying glass. The two Polaroid squares P1 and P2 are placed on either side of the
beaker as shown in the figure. The Polaroid squares are held crossed to each other so that the light only passes through when the medium between them is anisotropic and is cut off when the medium transforms to isotropic liquid.

(ii) Polarizing microscopy:

The ordinary usual optical method discussed above has certain limitations over its utility. Interstate transition occurring on account of polymesomorphism are rather difficult to be detected very accurately.

The transition temperatures were determined by using polarizing microscope “Leitz Labourlux 12 POL” (Wetzlar, Germany) with a heating stage.
A sample under investigation on polarizing microscope is required to be mounted on a slide. The slides were prepared by adopting following procedure:

The sample of a substance is taken on a cleaned and dried slide and was heated to a little above the melting temperature. At this stage coverslip was touched to the floating sample, pressed little and allowed to cool.

A substance is heated upto the isotropic liquid point and then a coverslip is pushed to touch liquid matter so that the sample substance spreads uniformly between coverslip and glass slide. On cooling the same, a thin film of sample substance appears between coverslip and slide which sticks firmly. This film was used for microscopic observations.

The calibration of microscope was checked by taking melting points and/or transition temperatures of very pure of known compounds.

Thus well prepared slides of a samples were placed on the heating stage of the polarizing microscope. The switch is put on and sample gets heated gradually at the rate of one degree per minute.

The determination of the various transition temperatures a glass slide, carrying a very thin film of the sample material with cover slip on it was observed under the hot stage polarizing
microscope. The temperature was raised at a rate of 5°C/min to find approximate transition temperatures. At this stage heating is stopped and cooling is permitted to take place until stable solid reappears. To determine the exact transition temperatures heating operation was repeated to still lower heating rate of about 1°C/min. The changing textures over the temperature ranges were carefully observed and recorded when the appearance of plane, focal conic, home tropic and threaded textures of smectic and nematic phases emerged under polarized light.

Appearance of focal conic structure and disappearance of cleavage lines of solid structure on heating is taken as solid-smectic transition. In order to confirm this change, the slide is disturbed with the help of spatula. The disturbed smectic phase structure can be seen indicating that it is not solid and the transition has actually taken place. Solid-nematic change is observed on heating with the appearance of threaded texture sharply at a definite temperature. All enantiotropic transitions are clearly detected on cooling the isotropic liquid, the reverse transitions takes place sharply at the same temperature or within ± 0.2 to ± 0.5°C. Isotropic-smectic change is marked by separation of small droplets from the isotropic liquid, which coalesce to give rise to a threaded region an unfailing characteristics of the nematic phase. Isotropic-smectic transition is indicated by appearance of batonnets which coalesce to form fine mosaic of focal conic pattern. Smectic-nematic changes are also clearly detected with a sharp variation in the texture from focal conic to threaded structure while heating and exactly the reversed sequence on cooling under the microscope.

Monotropic transitions are determined by carefully observing the isotropic liquid as it cools slowly until batonnets of smectic phase or droplets of nematic phase appear.

All the compounds under investigation were observed continuously under polarizing microscope from their solid state to isotropic liquid condition on heating and from isotropic liquid condition to solid stage on cooling ensuring conformation of all the transitions and making it sure that no transition gets unnoticed.

In case of doubt, the observations were repeated as many time as enough for clearing the doubt and in case the compounds were purified again and then were again under the microscope. In case of decomposition fresh slide was prepared each time for recording correct and accurate transition temperatures.
Table:-1

Transition temperatures of p-n-alkoxy benzoic acid

Transition temperature in °C

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
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<tbody>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>184.0</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>194.0</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>-</td>
<td>145.0</td>
<td>154.0</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>-</td>
<td>147.0</td>
<td>160.0</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>-</td>
<td>124.0</td>
<td>150.0</td>
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\[42\]
<table>
<thead>
<tr>
<th></th>
<th>Alkyl</th>
<th>cis-p-n-Alkoxy Cinnamic acid</th>
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<tr>
<td>6</td>
<td>Hexyl</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>Heptyl</td>
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</tr>
<tr>
<td>8</td>
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</tr>
<tr>
<td>9</td>
<td>Decyl</td>
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</tr>
<tr>
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<td>Dodecyl</td>
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</tr>
<tr>
<td>12</td>
<td>Hexadecyl</td>
<td>86.0</td>
</tr>
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</table>

Cis-p-n-Alkoxy Cinnamic acid [98]
Figure 8: Phase behavior of cis-p-n-alkoxy cinnamic acid
### 2.4 Homologous series-1

**Table-2 Transition temperatures in °C**

<table>
<thead>
<tr>
<th>Compound No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
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<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>172.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
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<td>Propyl</td>
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<td>Butyl</td>
<td>-</td>
<td>-</td>
<td>171.0</td>
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<td>Pentyl</td>
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<td>Hexyl</td>
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<td>Tetradecyl</td>
<td></td>
<td>143.0</td>
<td>162.0</td>
</tr>
<tr>
<td>11</td>
<td>Hexadecyl</td>
<td></td>
<td>145.0</td>
<td>154.0</td>
</tr>
</tbody>
</table>
Figure: 9 Phase behavior of homologous series-1
<table>
<thead>
<tr>
<th>No.</th>
<th>R=n-Alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{25}H_{19}O_{6}N</td>
<td>69.93</td>
<td>4.43</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{26}H_{21}O_{6}N</td>
<td>70.43</td>
<td>4.74</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{27}H_{23}O_{6}N</td>
<td>70.90</td>
<td>5.03</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{28}H_{25}O_{6}N</td>
<td>71.34</td>
<td>5.31</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{29}H_{27}O_{6}N</td>
<td>71.75</td>
<td>5.57</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{30}H_{32}O_{6}N</td>
<td>72.14</td>
<td>5.81</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{32}H_{33}O_{6}N</td>
<td>72.87</td>
<td>6.26</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{34}H_{37}O_{6}N</td>
<td>73.47</td>
<td>6.64</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{36}H_{41}O_{6}N</td>
<td>74.10</td>
<td>7.03</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{38}H_{45}O_{6}N</td>
<td>74.63</td>
<td>7.36</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{40}H_{49}O_{6}N</td>
<td>75.12</td>
<td>7.67</td>
</tr>
</tbody>
</table>
Table: 4 (a) IR Spectral Data

![Structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>1270 &gt;C=O of OC(<em>6)H(</em>{13})</td>
</tr>
<tr>
<td></td>
<td>1020, 1250, 1700 cm(^{-1}) -COO- group</td>
</tr>
<tr>
<td></td>
<td>810 p-substituted phenyl ring</td>
</tr>
<tr>
<td></td>
<td>695, 750 m-substituted phenyl ring</td>
</tr>
<tr>
<td></td>
<td>The above structure confirms by IR.</td>
</tr>
<tr>
<td>Tetradecyloxy</td>
<td>1175 &gt;C=O of OC(<em>{14})H(</em>{29}),</td>
</tr>
<tr>
<td></td>
<td>1025, 1250 &amp; 1600 ( -COO- group),</td>
</tr>
<tr>
<td></td>
<td>1530 (N=O stretching –NO(_2))</td>
</tr>
<tr>
<td></td>
<td>790 (m-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>2845 (Satu C-H str. of alkyl),</td>
</tr>
<tr>
<td></td>
<td>2915 C-H stre. of -CH=CH-, vinyl)</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by NMR.</td>
</tr>
</tbody>
</table>
NMR spectra Series-1
Table: 4(b) NMR Spectral Data

![Diagram of a molecule](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR spectra δ(ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decyloxy</td>
<td>1.272 (−CH$<em>2$ of OC$</em>{10}$H$<em>{21}$ group), 0.880 (s,−CH$<em>3$ of of OC$</em>{10}$H$</em>{21}$ group), 1.80 (conforms alkyl group of - OC$<em>{10}$H$</em>{21}$ group), 4.01 (t,−O−CH$<em>2$−CH$<em>2$ of - OC$</em>{10}$H$</em>{21}$ ), 6.93 and 8.05 (dd, p-substituted benzene ring), 7.26 (dd, m-substituted benzene ring), The structure confirms by NMR</td>
</tr>
<tr>
<td>Hexadecyloxy</td>
<td>0.873 (s, ,−CH$<em>3$ of of OC$</em>{16}$H$<em>{33}$ group), 1.250 (−CH$<em>2$ of OC$</em>{10}$H$</em>{21}$ group), 3.543 singlet(-OCH$_3$), 4.7385 broad (-CH=CH- group), 7.977 &amp; 8.122 (d, p-sub. phenyl ring), 8.630 &amp; 8.977 (dd, m-sub. phenyl ring) NMR confirms The structure confirms by NMR.</td>
</tr>
</tbody>
</table>
Table: 5 Texture

R= n-Alkyl chain

Texture: by miscibility method.

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dodecyloxy</td>
<td>Threaded nematic</td>
</tr>
<tr>
<td>Pentyloxy</td>
<td>Smectic-A type</td>
</tr>
</tbody>
</table>
2.5 Homologous series-2

Table: 6 Transition temperature in °C

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>200.0</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>206.0</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>-</td>
<td>-</td>
<td>190.0</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>-</td>
<td>-</td>
<td>201.0</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>-</td>
<td>142.0</td>
<td>170.0</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>-</td>
<td>124.0</td>
<td>160.0</td>
</tr>
<tr>
<td>7.</td>
<td>Heptyl</td>
<td></td>
<td>123.0</td>
<td>174.0</td>
</tr>
<tr>
<td>8.</td>
<td>Octyl</td>
<td>-</td>
<td>110.0</td>
<td>169.0</td>
</tr>
<tr>
<td>9.</td>
<td>Decyl</td>
<td>-</td>
<td>132.0</td>
<td>178.0</td>
</tr>
<tr>
<td>10.</td>
<td>Dodecyl</td>
<td>-</td>
<td>120.0</td>
<td>166.0</td>
</tr>
<tr>
<td>11.</td>
<td>Tetradecyl</td>
<td>-</td>
<td>125.0</td>
<td>152.0</td>
</tr>
<tr>
<td>12.</td>
<td>Hexadecyl</td>
<td>-</td>
<td>132.0</td>
<td>156.0</td>
</tr>
</tbody>
</table>
Figure 10: Phase behavior of homologous series 2
Table: 7 Elemental Analysis

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C\textsubscript{27}H\textsubscript{24}O\textsubscript{6}</td>
<td>72.97</td>
<td>5.40</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C\textsubscript{28}H\textsubscript{26}O\textsubscript{6}</td>
<td>73.36</td>
<td>5.68</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C\textsubscript{29}H\textsubscript{28}O\textsubscript{6}</td>
<td>73.73</td>
<td>5.93</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C\textsubscript{30}H\textsubscript{30}O\textsubscript{6}</td>
<td>74.07</td>
<td>6.17</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C\textsubscript{31}H\textsubscript{32}O\textsubscript{6}</td>
<td>74.40</td>
<td>6.40</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C\textsubscript{32}H\textsubscript{34}O\textsubscript{6}</td>
<td>74.71</td>
<td>6.61</td>
</tr>
<tr>
<td>7.</td>
<td>Heptyl</td>
<td>C\textsubscript{33}H\textsubscript{36}O\textsubscript{6}</td>
<td>75.00</td>
<td>6.82</td>
</tr>
<tr>
<td>8.</td>
<td>Octyl</td>
<td>C\textsubscript{34}H\textsubscript{38}O\textsubscript{6}</td>
<td>75.28</td>
<td>7.01</td>
</tr>
<tr>
<td>9.</td>
<td>Decyl</td>
<td>C\textsubscript{36}H\textsubscript{42}O\textsubscript{6}</td>
<td>75.79</td>
<td>7.37</td>
</tr>
<tr>
<td>10.</td>
<td>Dodecyl</td>
<td>C\textsubscript{38}H\textsubscript{46}O\textsubscript{6}</td>
<td>76.25</td>
<td>7.69</td>
</tr>
<tr>
<td>11.</td>
<td>Tetradecyl</td>
<td>C\textsubscript{40}H\textsubscript{50}O\textsubscript{6}</td>
<td>76.68</td>
<td>7.99</td>
</tr>
<tr>
<td>12.</td>
<td>Hexadecyl</td>
<td>C\textsubscript{42}H\textsubscript{54}O\textsubscript{6}</td>
<td>77.06</td>
<td>8.26</td>
</tr>
</tbody>
</table>
Table: 8 (a) IR Spectral Data

![Chemical Structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethyloxy</td>
<td>690 (Polymethylene-(CH₂)n-group of -OC₂H₅),</td>
</tr>
<tr>
<td></td>
<td>1170 (&gt;C=O group),</td>
</tr>
<tr>
<td></td>
<td>1260, 1605 (-COO- group),</td>
</tr>
<tr>
<td></td>
<td>990 (– CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>770 (1,2,4-trisubstituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>810 (p-sub. benzene ring)</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by IR</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>720 (polymethylene -(CH₂)n- group of OC₁₂H₂₅)</td>
</tr>
<tr>
<td></td>
<td>1165 (&gt;C=O),</td>
</tr>
<tr>
<td></td>
<td>1260, 1600 (-COO-),</td>
</tr>
<tr>
<td></td>
<td>960 (– CH=CH-)</td>
</tr>
<tr>
<td></td>
<td>845 (1,2,4-trisubstituted phenyl ring)</td>
</tr>
<tr>
<td></td>
<td>765 (p-substituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by IR spectra.</td>
</tr>
</tbody>
</table>
Current Data Parameters
NAME: Dec19-2012
EXPNO: 80
PROCNO: 1

F2 - Acquisition Parameters
Date: 20121219
Time: 15.55
INSTRUM: spect
PROBND: 5 mm PABBO BB-
FULPROG: zg30
TD: 65536
SOLVENT: DMSO
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 144
DW: 41.600 ussec
DE: 6.00 ussec
TE: 295.7 K
D1: 1.0000000 sec
TD0: 1

------ CHANNEL f1 ------
NUC1: 1H
P1: 10.90 ussec
PL1: -3.00 dB
SF01: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1299772 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

avatarsaifpu@yahoo.co.in
Current Data Parameters
NAME Dec19-2012
EXPN0 90
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121219
Time 16.00
INSTRUM spect
PROBHD 5 mm PABRO Bn-
PULFROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SMH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 228
DW 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

=== CHANNEL f1 ====
NUCI 1H
Pl 10.99 usec
PL1 -3.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299827 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME: Dec19-2012
EXPNO: 90
PROCNO: 1

F2 - Acquisition Parameters
Date: 20121219
Time: 16:00
INSTRUM: spect
PROBHD: 5 mm PABBO BB-PULPROG zg30
TD: 65536
SOLVENT: DMso
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 228
DW: 41.600 usec
DE: 6.00 usec
TE: 295.7 K
D1: 1.00000000 sec
TD0: 1

----------- CHANNEL f1 -----------
NUC1: 1H
P1: 10.00 usec
PL1: -3.00 dB
SF01: 400.1324710 MHz

F2 - Processing Parameters
SI: 32768
SF: 400.1299827 MHz
MDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
FC: 1.00

BRUKER AVANCE II 400 NMR Spectrometer
SAIF
Panjab University Chandigarh

avtar_saifpu@yahoo.co.in
Table: 8 (b) NMR Spectral Data

![Chemical Structure](image)

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>0.902 (triplet) (−CH₂ of OC₆H₁₃ group),&lt;br&gt;1.341 (t) (−CH₂−ₙ Polymethylene group of −OC₆H₁₃),&lt;br&gt;4.024 (Triplet) (−OCH₂−CH₂− of −OC₆H₁₃),&lt;br&gt;2.570 singlet (−OCH₃),&lt;br&gt;4.1008 broad (−CH=CH−),&lt;br&gt;6.899 &amp; 6.921 (−CO−CH=CH−),&lt;br&gt;7.897, 7.933 &amp; 8.923 (sub. phenyl ring)&lt;br&gt;NMR confirms the structure.</td>
</tr>
<tr>
<td>Decyloxy</td>
<td>0.878 (−CH₃ of OC₁₀H₂₁ group),&lt;br&gt;1.320 (t) (−CH₂−ₙ Polymethylene group of −OC₁₀H₂₁),&lt;br&gt;4.024 (Triplet) (−OCH₂−CH₂− of −OC₁₀H₂₁),&lt;br&gt;2.552 singlet (−OCH₃),&lt;br&gt;4.671 broad (−CH=CH−),&lt;br&gt;6.909 &amp; 6.930 (−CO−CH=CH−),&lt;br&gt;7.884, 7.905 &amp; 8.096r (sub. phenyl ring)&lt;br&gt;NMR confirms the structure.</td>
</tr>
</tbody>
</table>
Table: 9 Texture

\[
\begin{array}{c|c}
\text{Homologue} & \text{Texture} \\
\hline
\text{Pentyloxy} & \text{Threaded nematic} \\
\text{Tetradecyloxy} & \text{Schlieren nematic} \\
\end{array}
\]

\( R = \text{n-Alkyl chain} \)
2.6 Homologous series

Table: 10 Transition temperatures in $^\circ$C

![Chemical structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>152.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>170.0</td>
</tr>
<tr>
<td>3</td>
<td>Propyl</td>
<td>-</td>
<td>-</td>
<td>150.0</td>
</tr>
<tr>
<td>4</td>
<td>Butyl</td>
<td>-</td>
<td>-</td>
<td>165.0</td>
</tr>
<tr>
<td>5</td>
<td>Pentyl</td>
<td>140.0</td>
<td>150.0</td>
<td>176.0</td>
</tr>
<tr>
<td>6</td>
<td>Hexyl</td>
<td>102.0</td>
<td>122.0</td>
<td>178.0</td>
</tr>
<tr>
<td>7</td>
<td>Octyl</td>
<td>114.0</td>
<td>129.0</td>
<td>168.0</td>
</tr>
<tr>
<td>8</td>
<td>Decyl</td>
<td>112.0</td>
<td>128.0</td>
<td>164.0</td>
</tr>
<tr>
<td>9</td>
<td>Dodecyl</td>
<td>-</td>
<td>-</td>
<td>172.0</td>
</tr>
<tr>
<td>10</td>
<td>Tetradecyl</td>
<td>-</td>
<td>-</td>
<td>186.0</td>
</tr>
<tr>
<td>11</td>
<td>Hexadecyl</td>
<td>-</td>
<td>-</td>
<td>180.0</td>
</tr>
</tbody>
</table>
Figure: 11  Phase behavior of homologous series-3
**Table-11 Elemental Analysis**

![Chemical Structure](image.png)

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{25}H_{19}O_{6}N</td>
<td>69.93</td>
<td>4.43</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{26}H_{21}O_{6}N</td>
<td>70.43</td>
<td>4.74</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{27}H_{23}O_{6}N</td>
<td>70.90</td>
<td>5.03</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{28}H_{25}O_{6}N</td>
<td>71.34</td>
<td>5.31</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{29}H_{27}O_{6}N</td>
<td>71.75</td>
<td>5.57</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{30}H_{29}O_{6}N</td>
<td>72.14</td>
<td>5.81</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{32}H_{33}O_{6}N</td>
<td>72.87</td>
<td>6.26</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{34}H_{37}O_{6}N</td>
<td>73.47</td>
<td>6.64</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{36}H_{41}O_{6}N</td>
<td>74.10</td>
<td>7.03</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{38}H_{45}O_{6}N</td>
<td>74.63</td>
<td>7.36</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{40}H_{49}O_{6}N</td>
<td>75.12</td>
<td>7.67</td>
</tr>
</tbody>
</table>
IR spectra series-3
Table: 12 (a) IR Spectral Data

![Structural formula](image)

R = n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentyloxy</td>
<td>700 (-CH₂ₙ)-group of –OC₃H₁₁</td>
</tr>
<tr>
<td></td>
<td>1165 carbonil group,</td>
</tr>
<tr>
<td></td>
<td>1260,1610 &amp; 1680 -ester group,</td>
</tr>
<tr>
<td></td>
<td>920 (– CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>1430 &amp; 1510 (–NO₂),</td>
</tr>
<tr>
<td></td>
<td>845 (p-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>2875 (satu C-H str. of alkyl).</td>
</tr>
<tr>
<td></td>
<td>Above structure confirms by IR spectra.</td>
</tr>
<tr>
<td>Hexyloxy</td>
<td>700 (polymethylene -(CH₂)ₙ- group of OC₃H₁₃)</td>
</tr>
<tr>
<td></td>
<td>1140 (&gt;C=O),</td>
</tr>
<tr>
<td></td>
<td>1250,1610 &amp; 1700 (–COO-),</td>
</tr>
<tr>
<td></td>
<td>950 (– CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>1435 &amp; 1520 (–NO₂),</td>
</tr>
<tr>
<td></td>
<td>840 (p-substituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>Above structure confirms by IR spectra.</td>
</tr>
</tbody>
</table>
Current Data Parameters
NAME Feb16-2012
EXPN 560
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120216
Time 21.06
INSTRM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183999 Hz
AQ 2.7263477 sec
RG 144
DW 41.600 usec
DE 6.00 usec
TE 294.1 K
D1 1.0000000 sec
D2 1

-------- CHANNEL f1 --------
NUCL 1H
F1 10.90 usec
PL1 -3.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300946 MHz
WDM 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Bruker Avance II 400 NMR Spectrometer
SAIF Panjab University Chandigarh
avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME          Feb16-2012
EXNO          560
PROCNO        1

F2 - Acquisition Parameters
Date_          20120216
Time           21.06
INSTRUM        spect
PROBHD 5 mm PABBO BB-
FUPROG         zg30
TD            655.36
SOLVENT       DMSO
NS             8
DS             2
SWR           12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7263477 sec
RG            144
DW            41.600 usec
DE             6.00 usec
TE            294.1 K
D1            1.00000000 sec
TD0           1

====== CHANNEL f1 ======
NDC1           1H
F1             10.90 usec
PL1            -3.00 dB
SF01          400.1324710 MHz

F2 - Processing parameters
SI            32768
SF            400.1300946 MHz
WDW           EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME          Feb16-2012
EXNO          570
PROCNO         1

F2 - Acquisition Parameters
Date_          20120216
Time           21.11
INSTRUM        spec
PROBHD         5 mm FABBO BB-
FULP/WOG       zg30
TD             65536
SOLVENT        DMSO
NS             8
DS             2
SWH            12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7263477 sec
RG             64
DW             41.600 usec
DE             6.00 usec
TE             294.1 K
D1             1.000000000 sec
TD0            1

-------- CHANNEL f1 --------
NUC1          1H
F1            10.00 usec
PL1           -3.00 dB
SF01         400.1324710 MHz

F2 - Processing parameters
SI            32768
SF            400.1300996 MHz
WDW           HM
SSB           0
LB            0.30 Hz
GB            0
FC            1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Feb16-2012
EXPNO               570
PROCNO                1

F2 - Acquisition Parameters
Date_20120216
Time               21.11
INSTRUM            spect
PROBND            5 mm PABBO BB-
PULPROG          zg30
TD               65536
SOLVENT           DMSO
NS                8
DS                2
SWH          12019.230 Hz
FIDRES     0.183399 Hz
AQ         2.7263477 sec
RG               64
DW                41.600 usec
DE                6.00 usec
TE            294.1 K
D1           1.0000000 sec
TD0               1

======== CHANNEL f1 ========
NUC1          1H
P1          10.90 usec
PL1        -3.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI            32768
SP      400.1300996 MHz
WDW          EM
SSB            0
LB              0.30 Hz
GB            0
PC            1.00
Current Data Parameters
NAME                Feb16-2012
EXPMO               570
PROCNO              1

F2 - Acquisition Parameters
Date_{w}            20120216
Time                21.1
INSTRUM             spect
PROBHD              5 mm PABBO BB-
PULPROG             zg30
TD                  65536
SOLVENT             DMSO
NS                  8
DS                  2
SWH                 12019.230 Hz
FIDRES              0.183399 Hz
AQ                  2.7263477 sec
RG                  64
DW                  41.600 usec
DE                  6.00 usec
TE                  294.1 K
D1                  1.00000000 sec
TD0                 1

-------- CHANNEL f1 --------
NUC1                1H
F1                  10.90 usec
FL1                 -3.00 dB
SF01                400.1324710 MHz

F2 - Processing parameters
SI                   32768
SF                  400.1300996 MHz
WDM                 EM
SSB                  0
LB                  0.30 Hz
GB                  0
PC                  1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Feb16-2012
EXPNO        570
PROCNO       1
F2 - Acquisition Parameters
Date_         20120216
Time          21:11
INSTRUM       spect
PULPROG      zg30
TD            65536
SOLVENT       DMSO
NS            8
DS            2
SWH           12019.230 Hz
FIDRES        0.183399 Hz
AQ            2.7263477 sec
RG            64
DW            41.600 usec
DE            6.00 usec
TE            294.1 K
TD0           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.90 usec
PL1               -3.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF               400.1300996 MHz
WDW             EM
SSB              0
LB              0.30 Hz
GB              0
PC              1.00

avtar_saifpu@yahoo.co.in
Table: 12 (b) NMR Spectral Data

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Octyloxy</td>
<td>0.950 (−CH$_3$ of OC$<em>8$H$</em>{17}$ group), 1.122 (−CH$_2$)$_n$ Polymethylene group of −OC$<em>8$H$</em>{17}$), 3.690 (Triplet) (−OCH$_2$−CH$_2$ of −OC$<em>8$H$</em>{17}$), 5.077 &amp; 5.121 Broad (−CH=CH−), 6.557 &amp; 6.579 (−CH=CH-CO−), 7.483, 7.058, &amp; 7.607 (p-sub. benzene ring) The structure confirms by NMR spectra.</td>
</tr>
<tr>
<td>Decyloxy</td>
<td>0.959 (−CH$<em>3$ of OC$</em>{10}$H$_{21}$ group), 1.179 (−CH$<em>2$)$<em>n$ Polymethylene group of −OC$</em>{10}$H$</em>{21}$), 3.698 (Triplet) (−OCH$<em>2$−CH$<em>2$ of −OC$</em>{10}$H$</em>{21}$), 6.579 &amp; 6.601 (−CH=CH-CO− group), 6.579, 7.608 &amp; 7.630 (p-substituted phenyl ring) The structure confirms by NMR spectra.</td>
</tr>
</tbody>
</table>
Table: 13 Texture

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>Threaded nematic</td>
</tr>
<tr>
<td>Octyloxy</td>
<td>Schlieren nematic</td>
</tr>
<tr>
<td>Pentyloxy</td>
<td>Smectic-A type</td>
</tr>
</tbody>
</table>
2.7 Homologous series-4

Table: 14 Transition temperatures in °C

![Chemical structure of the compound](image)

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>185.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>161.0</td>
</tr>
<tr>
<td>3</td>
<td>Propyl</td>
<td>-</td>
<td>164.0</td>
<td>215.0</td>
</tr>
<tr>
<td>4</td>
<td>Butyl</td>
<td>-</td>
<td>-</td>
<td>192.0</td>
</tr>
<tr>
<td>5</td>
<td>Pentyl</td>
<td>-</td>
<td>152.0</td>
<td>182.0</td>
</tr>
<tr>
<td>6</td>
<td>Hexyl</td>
<td>-</td>
<td>124.0</td>
<td>175.0</td>
</tr>
<tr>
<td>7</td>
<td>Octyl</td>
<td>-</td>
<td>120.0</td>
<td>182.0</td>
</tr>
<tr>
<td>8</td>
<td>Decyl</td>
<td>-</td>
<td>109.0</td>
<td>163.0</td>
</tr>
<tr>
<td>9</td>
<td>Dodecyl</td>
<td>-</td>
<td>114.0</td>
<td>155.0</td>
</tr>
<tr>
<td>10</td>
<td>Tetradecyl</td>
<td>-</td>
<td>112.0</td>
<td>158.0</td>
</tr>
<tr>
<td>11</td>
<td>Hexadecyl</td>
<td>-</td>
<td>-</td>
<td>158.0</td>
</tr>
</tbody>
</table>
Figure 12: Phase behavior of homologous series - 4
<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{25}H_{19}O_{6}N</td>
<td>69.93</td>
<td>4.43</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{26}H_{21}O_{6}N</td>
<td>70.43</td>
<td>4.74</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{27}H_{23}O_{6}N</td>
<td>70.90</td>
<td>5.03</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{28}H_{25}O_{6}N</td>
<td>71.34</td>
<td>5.31</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{29}H_{27}O_{6}N</td>
<td>71.75</td>
<td>5.57</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{30}H_{29}O_{6}N</td>
<td>72.14</td>
<td>5.81</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{32}H_{33}O_{6}N</td>
<td>72.87</td>
<td>6.26</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{34}H_{37}O_{6}N</td>
<td>73.47</td>
<td>6.64</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{36}H_{41}O_{6}N</td>
<td>74.10</td>
<td>7.03</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{38}H_{45}O_{6}N</td>
<td>74.63</td>
<td>7.36</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{40}H_{49}O_{6}N</td>
<td>75.12</td>
<td>7.67</td>
</tr>
</tbody>
</table>
IR Spectra of Series-4
Table: 16(a) IR Spectral Data

![Chemical Structure](attachment:image.png)

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>625 (Polymethylene-(CH₂)n-group of –OC₆H₁₃)</td>
</tr>
<tr>
<td></td>
<td>1160 &gt;C=O,</td>
</tr>
<tr>
<td></td>
<td>1230,1600 &amp; 1680 ester,</td>
</tr>
<tr>
<td></td>
<td>930 – CH=CH₂,</td>
</tr>
<tr>
<td></td>
<td>1415 ¹ (–NO₂ group),</td>
</tr>
<tr>
<td></td>
<td>760 (m-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>IR confirms above structure</td>
</tr>
<tr>
<td>Decyloxy</td>
<td>625 (polymethylene-(CH₂)n-group of OC₁₀H₂₁)</td>
</tr>
<tr>
<td></td>
<td>1160 (&gt;C=O group),</td>
</tr>
<tr>
<td></td>
<td>1230,1600 &amp; 1680 (–COO -group),</td>
</tr>
<tr>
<td></td>
<td>930 (– CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>1415 &amp; 1430 (–NO₂ group),</td>
</tr>
<tr>
<td></td>
<td>820 (p-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>770 (m-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>IR confirms above structure</td>
</tr>
</tbody>
</table>
Current Data Parameters
NAME    Feb16-2012
EXPNO   580
PROCNO  1

F2 - Acquisition Parameters
Date_   20120216
Time    21.16
INSTRUM spect
FREQMD  5 mm PARBO BB-
PULPROG zg90
TD      65536
SOLVENT DMSO
MS      8
DS      2
SWM     12019.230 Hz
FIDRES  0.183399 Hz
AQ      2.7263477 sec
RG      161
DW      41.600 usec
DE      6.00 usec
TE      294.1 K
D1      1.00000000 sec
TD0     1

-------- CHANNEL f1 --------

F2 - Processing parameters
SI      32768
SF      400.1300945 MHz
NDM    20
SSB     0
LB      0.30 Hz
GB      0
PC      1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME    Feb16-2012
EXPDNO  580
PROCNO  1

F2 - Acquisition Parameters
Date_  20120216
Time   21.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
FIDNAME zg30
TD      65536
SOLVENT DMSO
NS      8
DS      2
SWH    12019.230 Hz
FIDRES 0.183399 Hz
AQ      2.7263477 sec
RG      161
DW      41.600 usec
DE      6.00 usec
TE      294.1 K
D1      1.0000000 sec
TD0     1

--- CHANNEL f1 ---
NUC1    1H
PL1    -3.00 dB
SF01   400.1324710 MHz

F2 - Processing parameters
SI      32768
SF      400.1300945 MHz
WDW     EM
SSB     0
LB      0.30 Hz
PC      1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME        Feb16-2012
EXPNO       590
PROCNO      1

F2 - Acquisition Parameters
Date_        20120216
Time         21:21
INSTRUM      spect
PROBHD 5 mm PABBO BB-
PULPROM      zg30
TD           65536
SOLVENT      DMSO
NS           8
DS           2
SWH          12019.230 Hz
FIDRES       0.183399 Hz
AQ           2.7263477 sec
RG           57
DW           41.600 usec
DE           6.000 usec
TE           294.1 K
D1           1.00000000 sec
TD0          1

---------- CHANNEL f1 ----------
NUC1         1H
F1           10.90 usec
PL1          -3.00 dB
SF01         400.1324710 MHz

F2 - Processing parameters
SI            32768
SF            400.1299738 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

avtar_saifpu@yahoo.co.in
BRUKER AVANCE II 400 NMR Spectrometer
SAIF Panjab University Chandigarh

Current Data Parameters
NAME Feb16-2012
EXFNO 590
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120216
Time 21.21
INSTRUM spect
PROBD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DE 2
SWH 12019.230 Hz
FIDRES 0.183799 Hz
AQ 2.7263477 sec
RG 57
DW 41.600 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.90 usec
PH1 -3.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299738 Hz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in
Table: 16 (b) NMR Spectral Data

![NMR Spectral Data Diagram]

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Octyloxy</td>
<td>0.993 (−CH₃ of OC₈H₁₇ group), 1.173 (−CH₂ₙ Polymethylene group of −OC₈H₁₇), 3.718 (Triplet) (−OCH₂CH₂ of −OC₈H₁₇), 3.223 broad (−CH=CH−), 6.611 &amp; 6.579 (−CH=CH-CO−), 6.633 (p-sub. benzene ring) 7.60 &amp; 7.734 (p-sub. benzene ring) The structure confirms by NMR spectra.</td>
</tr>
<tr>
<td>Tetradecyloxy</td>
<td>0.872 (−CH₃ of OC₁₀H₂₁ group), 1.253 (−CH₂ₙ Polymethylene group of −OC₁₀H₂₁), 4.000 (Triplet) (−OCH₂CH₂ of −OC₁₀H₂₁), 4.229 &amp; 4.300 (−CH=CH-CO−), 6.88, &amp; 7.931 (p-sub. benzene ring), 6.90 (m-sub. benzene ring), The structure confirms by NMR spectra.</td>
</tr>
</tbody>
</table>

Table: 17 Texture
Texture of mesophase were determined by miscibility method.

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentyloxy</td>
<td>Threaded Nematic</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>Schlieren</td>
</tr>
</tbody>
</table>
2.8 Homologous series-5

Table: 18 Transition temperatures in °C

R= n-Alkyl chain

Transition temp. in °C

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>183.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>160.0</td>
</tr>
<tr>
<td>3</td>
<td>Propyl</td>
<td>-</td>
<td>-</td>
<td>169.0</td>
</tr>
<tr>
<td>4</td>
<td>Butyl</td>
<td>-</td>
<td>-</td>
<td>161.0</td>
</tr>
<tr>
<td>5</td>
<td>Pentyl</td>
<td>-</td>
<td>-</td>
<td>170.0</td>
</tr>
<tr>
<td>6</td>
<td>Hexyl</td>
<td>116.0</td>
<td>134.0</td>
<td>165.0</td>
</tr>
<tr>
<td>7</td>
<td>Octyl</td>
<td>99.0</td>
<td>112.0</td>
<td>155.0</td>
</tr>
<tr>
<td>8</td>
<td>Decyl</td>
<td>109.0</td>
<td>125.0</td>
<td>172.0</td>
</tr>
<tr>
<td>9</td>
<td>Dodecyl</td>
<td>106.0</td>
<td>136.0</td>
<td>158.0</td>
</tr>
<tr>
<td>10</td>
<td>Tetradecyl</td>
<td>-</td>
<td>105.0</td>
<td>120.0</td>
</tr>
<tr>
<td>11</td>
<td>Hexadecyl</td>
<td>-</td>
<td>110.0</td>
<td>122.0</td>
</tr>
</tbody>
</table>
Figure 13: Phase behavior of homologous series - 5
Table: Elemental Analysis

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{23}H_{17}O_{6}N</td>
<td>68.49</td>
<td>4.22</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{24}H_{19}O_{6}N</td>
<td>69.06</td>
<td>4.56</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{25}H_{21}O_{6}N</td>
<td>69.60</td>
<td>4.87</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{26}H_{23}O_{6}N</td>
<td>70.11</td>
<td>5.17</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{27}H_{25}O_{6}N</td>
<td>70.59</td>
<td>5.45</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{28}H_{27}O_{6}N</td>
<td>71.03</td>
<td>5.71</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{30}H_{31}O_{6}N</td>
<td>71.86</td>
<td>6.19</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{32}H_{35}O_{6}N</td>
<td>72.59</td>
<td>6.62</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{34}H_{39}O_{6}N</td>
<td>73.25</td>
<td>7.00</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{36}H_{43}O_{6}N</td>
<td>73.85</td>
<td>7.35</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{38}H_{47}O_{6}N</td>
<td>74.39</td>
<td>7.67</td>
</tr>
</tbody>
</table>
Table: 20(a) IR Spectral Data

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm(^{-1})</th>
</tr>
</thead>
</table>
| Hexyloxy  | 720 (Polymethylene-(CH\(_2\))\(_n\))-group of –OC\(_6\)H\(_{13}\))  
           | 1160 (>C=O),  
           | 1250 & 1650 \(^1\) (-COO-),  
           | 950 (–CH=CH),  
           | 1380 & 1525 (–NO\(_2\) group),  
           | 680 & 760 (m-sub. phenyl ring),  
           | The above structure confirms by IR spectra. |
| Decyloxy  | 720 (polymethylene -(CH\(_2\))\(_n\)- of OC\(_{10}\)H\(_{21}\))  
           | 1150 (>C=O),  
           | 1250, 1600 & 1660 \(^1\) (–COO group),  
           | 950 (–CH=CH-),  
           | 1440 & 1500 (–NO\(_2\) group),  
           | 830 (pera-sub. phenyl ring),  
           | 640 & 760 (m-sub. phenyl ring),  
           | The above structure confirms by IR spectra. |
Current Data Parameters
NAME           Feb16-2012
EXPNO          600
PROCNO         1

F2 - Acquisition Parameters
Date_           20120216
Time            21.26
INSTRUM         spect
PROBHDL         5 mm PABBO BB-
PULFROG         zg30
TD              65536
SOLVENT         DMSO
NS              8
DS              2
SWH             12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7263477 sec
RG              144
DW              41.600 usec
DE              6.00 usec
TE              294.1 K
D1              1.0000000 sec
TDO             1

======== CHANNEL f1 ========
NUC1            1H
P1              10.90 usec
PL1             -3.00 dB
SFO1            400.1324710 MHz

F2 - Processing parameters
SI              32768
SF              400.1299782 MHz
WDW             EM
SSB             0
LB              0.30 Hz
GB              0
PC              1.00

DMS-5
Current Data Parameters
NAME: Feb16-2012
EXNO: 600
PROCNO: 1

F2 - Acquisition Parameters
Date: 2012.02.16
Time: 21:26
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
PULPROG: zg30
TD: 65536
SOLVENT: DMSO
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 144
DW: 41.600 usec
DE: 6.00 usec
TE: 294.1 K
D1: 1.0000000 sec
TD0: 1

--- CHANNEL f1 ---
NUC: 1H
P1: 10.90 usec
PL1: -3.00 dB
SF01: 400.1324710 MHz
SF: 32768
SF: 400.1299782 MHz
WDM: EM
SSB: 0
LB: 0.30 Hz
NE: 1.00
Current Data Parameters
NAME      Feb16-2012
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date       20120216
Time       21.31
INSTRUM    spect
PROBHD     5 mm PABBO BB-
PULPROG     zg30
TD          65536
SOLVENT    DMSO
NS          8
DS          2
SWH        12019.230 Hz
FDRES      0.183399 Hz
AQ          2.7263477 sec
RG          144
DW          41.600 usec
DE          6.00 usec
TE          294.1 K
DI          1.0000000 sec
TOO         1

====== CHANNEL F1 ======
NUCL       1H
F1         10.90 usec
PL1        -3.00 dB
SPOL       400.1324710 MHz

F2 - Processing parameters
SI          32768
SF          400.1299774 MHz
WDW         EM
SBB         0
LB          0.30 Hz
GB          0
PC          1.00

avatar_saifpu@yahoo.co.in
Current Data Parameters

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
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<tr>
<td>PROCNO</td>
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<td>F2 - Acquisition Parameters</td>
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<tr>
<td>Date_</td>
<td>20120216</td>
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<tr>
<td>Time</td>
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</tr>
<tr>
<td>INSTRUM</td>
<td>spect</td>
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<tr>
<td>PROBHD</td>
<td>5 mm PABBO BB-</td>
</tr>
<tr>
<td>PULPSPG</td>
<td>zg30</td>
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<td>TD</td>
<td>65336</td>
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<tr>
<td>SOLVENT</td>
<td>DMSO</td>
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<tr>
<td>NS</td>
<td>8</td>
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<tr>
<td>DS</td>
<td>2</td>
</tr>
<tr>
<td>SWH</td>
<td>12019.230 Hz</td>
</tr>
<tr>
<td>FIDRES</td>
<td>0.183399 Hz</td>
</tr>
<tr>
<td>AQ</td>
<td>2.7263477 sec</td>
</tr>
<tr>
<td>RG</td>
<td>144</td>
</tr>
<tr>
<td>DW</td>
<td>41.600 usec</td>
</tr>
<tr>
<td>DE</td>
<td>6.00 usec</td>
</tr>
<tr>
<td>TE</td>
<td>294.1 K</td>
</tr>
<tr>
<td>DI</td>
<td>1.00000000 sec</td>
</tr>
<tr>
<td>TD0</td>
<td>1</td>
</tr>
</tbody>
</table>

====== CHANNEL f1 ======

| NUC1         | 1H         |
| P1           | 10.90 usec |
| PL1          | -3.00 dB   |
| SF01         | 400.1324710 MHz |

F2 - Processing parameters

| SI           | 32768      |
| SF           | 400.1299774 MHz |

avtar_saifpu@yahoo.co.in
**Table:-20(b) NMR Spectral Data**

![Structure](https://via.placeholder.com/150)

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Octyloxy</td>
<td>0.880 (−CH₃ of OC₈H₁₇ group), 1.307 (Triplet) (−CH₂)n Polymethylene group of −OC₈H₁₇), 4.000 (Triplet) (−OCH₂−CH₂− of −OC₈H₁₇), 6.920 broad (−CH=CH- group), 7.29 (-CH=CH-CO-group), 6.75 (p-sub.benzene ring), 8.10 (p-sub.benzene ring), NMR confirms the structure.</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>0.871 (−CH₃ of OC₁₂H₂₅ group), 1.25 &amp; 1.76 (−CH₂)n Polymethylene group of −OC₁₂H₂₅), 4.002 (Triplet) (−OCH₂−CH₂− of −OC₁₂H₂₅), 7.2 &amp; 7.00 (broad) (-CH=CH-CO-group), 8.1 (p-substituted phenyl ring), 6.90 (m-substituted phenyl ring), NMR confirms the structure.</td>
</tr>
</tbody>
</table>
Table: 21 Texture

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>Threaded Nematic</td>
</tr>
<tr>
<td>Tetra decyloxy</td>
<td>Schlieren</td>
</tr>
</tbody>
</table>
### 2.9 Homologous series-6

#### Table: Transition temperatures in °C

![Chemical structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>169.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
<td>-</td>
<td>134.0</td>
<td>167.0</td>
</tr>
<tr>
<td>3</td>
<td>Propyl</td>
<td>-</td>
<td>153.0</td>
<td>160.0</td>
</tr>
<tr>
<td>4</td>
<td>Butyl</td>
<td>-</td>
<td>140.0</td>
<td>164.0</td>
</tr>
<tr>
<td>5</td>
<td>Pentyl</td>
<td>-</td>
<td>145.0</td>
<td>158.0</td>
</tr>
<tr>
<td>6</td>
<td>Hexyl</td>
<td>110.0</td>
<td>117.0</td>
<td>166.0</td>
</tr>
<tr>
<td>7</td>
<td>Heptyl</td>
<td>102.0</td>
<td>115.0</td>
<td>167.0</td>
</tr>
<tr>
<td>8</td>
<td>Octyl</td>
<td>104.0</td>
<td>118.0</td>
<td>168.0</td>
</tr>
<tr>
<td>9</td>
<td>Decyl</td>
<td>92.0</td>
<td>120.0</td>
<td>159.0</td>
</tr>
<tr>
<td>10</td>
<td>Dodecyl</td>
<td>95.0</td>
<td>119.0</td>
<td>140.0</td>
</tr>
<tr>
<td>11</td>
<td>Tetradecyl</td>
<td>90.0</td>
<td>105.0</td>
<td>138.0</td>
</tr>
<tr>
<td>12</td>
<td>Hexadecyl</td>
<td>-</td>
<td>100.0</td>
<td>136.0</td>
</tr>
</tbody>
</table>
Figure 14: Phase behavior of homologous series-6
Table: 23 Elemental Analysis

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1. Methyl</td>
<td></td>
<td>C_{25}H_{22}O_6</td>
<td>71.77</td>
<td>5.26</td>
</tr>
<tr>
<td>2. Ethyl</td>
<td></td>
<td>C_{26}H_{24}O_6</td>
<td>72.22</td>
<td>5.56</td>
</tr>
<tr>
<td>3. Propyl</td>
<td></td>
<td>C_{27}H_{26}O_6</td>
<td>72.65</td>
<td>5.83</td>
</tr>
<tr>
<td>4. Butyl</td>
<td></td>
<td>C_{28}H_{30}O_6</td>
<td>73.04</td>
<td>6.09</td>
</tr>
<tr>
<td>5. Pentyl</td>
<td></td>
<td>C_{29}H_{32}O_6</td>
<td>73.42</td>
<td>6.33</td>
</tr>
<tr>
<td>6. Hexyl</td>
<td></td>
<td>C_{30}H_{34}O_6</td>
<td>73.77</td>
<td>6.56</td>
</tr>
<tr>
<td>7. Heptyl</td>
<td></td>
<td>C_{31}H_{36}O_6</td>
<td>74.10</td>
<td>6.77</td>
</tr>
<tr>
<td>8. Octyl</td>
<td></td>
<td>C_{32}H_{38}O_6</td>
<td>74.42</td>
<td>6.98</td>
</tr>
<tr>
<td>9. Decyl</td>
<td></td>
<td>C_{34}H_{42}O_6</td>
<td>75.00</td>
<td>7.35</td>
</tr>
<tr>
<td>10. Dodecyl</td>
<td></td>
<td>C_{36}H_{46}O_6</td>
<td>75.52</td>
<td>7.69</td>
</tr>
<tr>
<td>11. Tetradecyl</td>
<td></td>
<td>C_{38}H_{50}O_6</td>
<td>76.00</td>
<td>8.00</td>
</tr>
<tr>
<td>12. Hexadecyl</td>
<td></td>
<td>C_{40}H_{54}O_6</td>
<td>76.43</td>
<td>8.28</td>
</tr>
</tbody>
</table>
### Table: 24 (a) Spectral Data

R = n-Alkyl chain

![Chemical Structure](image)

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm(^{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Hexyloxy</strong></td>
<td>625 (Polymethylene-(CH(_2))(_n)-group of (-OC(<em>6)H(</em>{13})), 1170 (&gt;C=O group), 1250 &amp; 1605 (-COO- group), 940 (– CH=CH- group), 770 (p-sub. benzene ring), 820 (1,2,4-trisubstituted benzene ring), The above structure confirms by IR.</td>
</tr>
<tr>
<td><strong>Octyloxy</strong></td>
<td>645 (polymethylene -(CH(_2))(_n)- group of OC(<em>8)H(</em>{17})), 1150 (&gt;C=O), 1260 &amp; 1605 (-COO), 960 (– CH=CH-), 845 (1,2,4-trisubstituted phenyl ring) 730 (p-substituted phenyl ring), The above structure confirms by IR.</td>
</tr>
</tbody>
</table>
NMR spectra of Series-6

Current Data Parameters
NAME Dec19-2012
EXFNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121219
Time 15.50
INSTRUM spect
PROBHD 5 mm PARBO BB-
FULTROG zg10
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWM 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 203
DW 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.0000000 sec
T0 1

CHANNEL f1
NUC 1H
F1 10.90 usec
PL1 -3.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299806 MHz
WDW EM
SMB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME Dec19-2012
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121219
Time 15.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
FIDPROG zg30
TD 653.36
SOLVENT DMSO
NS 8
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 203
DW 41.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.0000000 sec
TDO 1

-------- CHANNEL f1 ---------
NUCL 1H
P1 10.90 usec
PL1 -3.00 dB
SPOL 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299806 MHz
WDW 1M
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in
| ppm | 0.0001 | 0.8544 | 0.8718 | 0.8874 | 1.1257 | 1.2557 | 1.3086 | 1.4281 | 1.4458 | 1.7336 | 1.7496 | 1.7680 | 1.7852 | 1.8007 | 2.1256 | 2.5692 | 3.9908 | 4.0067 | 4.0225 | 4.9840 | 6.8996 | 6.9207 | 7.8930 | 7.9140 | 8.0267 | 8.0674 | 8.9545 |
|-----|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|

**Current Data Parameters**

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<td>EXPNO</td>
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</tr>
<tr>
<td>PROCNO</td>
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<td>Time</td>
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<td>PROBHD</td>
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<td>TD</td>
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<tr>
<td>SOLVENT</td>
<td>DMSO</td>
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<tr>
<td>MS</td>
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<td>RG</td>
<td>71.8</td>
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<tr>
<td>DW</td>
<td>41.600 usec</td>
</tr>
<tr>
<td>DE</td>
<td>6.00 usec</td>
</tr>
<tr>
<td>TE</td>
<td>295.7 K</td>
</tr>
<tr>
<td>D1</td>
<td>1.00000000 sec</td>
</tr>
<tr>
<td>TD0</td>
<td>1</td>
</tr>
</tbody>
</table>

**CHANNEL f1**

| NUC1       | 1H               |
| F1         | 10.90 usec       |
| PL1        | -3.00 dB         |
| SFO1       | 400.1324710 MHz  |

**F2 - Processing parameters**

| SI          | 32768            |
| SF          | 400.1299746 MHz  |
| WDW         | 3M               |
| SSB         | 0                |
| LB          | 0.30 Hz          |
| GB          | 0                |
| PC          | 1.00             |

---

**BRUKER AVANCE II 400 NMR Spectrometer**

**SAIF**

Panjab University Chandigarh

avtar_saifpu@yahoo.co.in
Table: 24(b) NMR Spectral Data

R = n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Propyloxy</td>
<td>0.870 (−CH$_3$ of OC$_3$H$_7$ group), 1.029 (Triplet) (−CH$_2$)$_n$ Polymethylene group of −OC$_3$H$_7$), 3.980 (Triplet) (−OCH$_2$−CH$_2$ −OC$_3$H$_7$), 4.075 broad (−CH=CH- group), 3.964 (singlet) (−OCH$_3$ group), 6.911 &amp; 6.933 (−CO−CH=CH− group), 7.892, 7.913 &amp; 8.063 (substituted phenyl ring), NMR confirms the structure.</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>0.871 (−CH$<em>3$ of OC$</em>{12}$H$_{25}$ group), 1.255 (−CH$<em>2$)$<em>n$ Polymethylene group of −OC$</em>{12}$H$</em>{25}$), 4.067 (Triplet) (−OCH$<em>2$−CH$<em>2$ −OC$</em>{12}$H$</em>{25}$), 6.899 &amp; 7.920 (broad) (−CO−CH=CH− group), 4.984 broad (−CH=CH- group), 3.990 (singlet) (−OCH$_3$ group), 7.893, 7.914 &amp; 8.954 (substituted phenyl ring) NMR confirms the structure</td>
</tr>
</tbody>
</table>
Table: Texture

R=n-alkyl chain

Texture: by miscibility method.

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>Smectic-A</td>
</tr>
<tr>
<td>Butyloxy</td>
<td>Threaded Nematic</td>
</tr>
<tr>
<td>Hexadecyloxy</td>
<td>Schlieren Nematic</td>
</tr>
</tbody>
</table>
### 2.10 Homologous series-7

#### Table: 26 Transition temperatures in °C

![Chemical structure](image)

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>R= n-Alkyl group</th>
<th>Smectic</th>
<th>Nematic</th>
<th>Isotropic</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Methyl</td>
<td>-</td>
<td>-</td>
<td>191.0</td>
</tr>
<tr>
<td>2</td>
<td>Ethyl</td>
<td>-</td>
<td>-</td>
<td>200.0</td>
</tr>
<tr>
<td>3</td>
<td>Propyl</td>
<td>-</td>
<td>146.0</td>
<td>164.0</td>
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<tr>
<td>4</td>
<td>Butyl</td>
<td>-</td>
<td>120.0</td>
<td>140.0</td>
</tr>
<tr>
<td>5</td>
<td>Pentyl</td>
<td>-</td>
<td>110.0</td>
<td>160.0</td>
</tr>
<tr>
<td>6</td>
<td>Hexyl</td>
<td>114.0</td>
<td>137.0</td>
<td>167.0</td>
</tr>
<tr>
<td>7</td>
<td>Octyl</td>
<td>96.0</td>
<td>120.0</td>
<td>169.0</td>
</tr>
<tr>
<td>8</td>
<td>Decyl</td>
<td>100.0</td>
<td>120.0</td>
<td>166.0</td>
</tr>
<tr>
<td>9</td>
<td>Dodecyl</td>
<td>94.0</td>
<td>129.0</td>
<td>162.0</td>
</tr>
<tr>
<td>10</td>
<td>Tetradecyl</td>
<td>96.0</td>
<td>122.0</td>
<td>136.0</td>
</tr>
<tr>
<td>11</td>
<td>Hexadecyl</td>
<td>-</td>
<td>110.0</td>
<td>128.0</td>
</tr>
</tbody>
</table>
Figure 15: Phase behavior of homologous series-7
Table: 27 Elemental Analysis

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{23}H_{17}O_6N</td>
<td>68.49</td>
<td>4.22</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{24}H_{19}O_6N</td>
<td>69.06</td>
<td>4.56</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{25}H_{21}O_6N</td>
<td>69.60</td>
<td>4.87</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{26}H_{23}O_6N</td>
<td>70.11</td>
<td>5.17</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{27}H_{25}O_6N</td>
<td>70.59</td>
<td>5.45</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{28}H_{27}O_6N</td>
<td>71.03</td>
<td>5.71</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{30}H_{31}O_6N</td>
<td>71.86</td>
<td>6.19</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{32}H_{35}O_6N</td>
<td>72.59</td>
<td>6.62</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{34}H_{39}O_6N</td>
<td>73.25</td>
<td>7.00</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{36}H_{43}O_6N</td>
<td>73.85</td>
<td>7.35</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{38}H_{47}O_6N</td>
<td>74.39</td>
<td>7.67</td>
</tr>
</tbody>
</table>
### Table: 28 (a) IR Spectral Data

![Structural Diagram](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>730 (Polymethylene-(CH₂)n-group of –OC₆H₁₃)</td>
</tr>
<tr>
<td></td>
<td>1040, 1220 &amp; 1690 (-COO- group),</td>
</tr>
<tr>
<td></td>
<td>660 (–CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>1510 (–NO₂ group),</td>
</tr>
<tr>
<td></td>
<td>830 (p-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>2900 (alkyl group)</td>
</tr>
<tr>
<td></td>
<td>The above structure confirms by IR.</td>
</tr>
<tr>
<td>Propyloxy</td>
<td>620 (polymethylene -(CH₂)n- group of OC₃H₇)</td>
</tr>
<tr>
<td></td>
<td>1165 (&gt;C=O),</td>
</tr>
<tr>
<td></td>
<td>1225, 1600 &amp; 1680 (–COO group),</td>
</tr>
<tr>
<td></td>
<td>965 (–CH=CH- group),</td>
</tr>
<tr>
<td></td>
<td>1415 (–NO₂ group),</td>
</tr>
<tr>
<td></td>
<td>845 (p-substituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>The above structure confirms by IR.</td>
</tr>
</tbody>
</table>
NMR Spectra of Series-7

Current Data Parameters
NAME: Feb16-2012
EXPNO: 21
PROCNO: 1

F2 - Acquisition Parameters
Date: 20120216
Time: 21.36
INSTRUM: spect
PROBNO: 5 mm PABBO BB-
FULFROG: zg30
TD: 65536
SOLVENT: DMSO
NS: 8
DG: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7264777 sec
RG: 144
DW: 41.600 usec
DE: 6.00 usec
TE: 294.0 K
D1: 1.00000000 sec
TD0: 1

-------- CHANNEL f1 --------
NUC1: 1H
F1: 10.90 usec
PL1: -3.00 dB
SF01: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SP: 400.12399730 MHz
MDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

avtar_saifpu@yahoo.co.in
BRUKER AVANCE II 400 NMR Spectrometer
SAIF Panjab University Chandigarh

Current Data Parameters
NAME Feb16-2012
EXPNO 21
PROCN1 1

F2 - Acquisition Parameters
Date_ 20120216
Time 21.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DE 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 144
DW 41.600 usec
DE 6.00 usec
TE 294.0 K
DI 1.00000000 sec
TDO 1

-------- CHANNEL f1 --------
NUCI 1H
P1 10.90 usec
PL1 -3.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1299730 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avatar_saifpu@yahoo.co.in
Current Data Parameters
NAME          Feb16-2012
EXPNO         31
PROCNO        1

F2 - Acquisition Parameters
Date_          20120216
Time           21.41
INSTRUM        spect
PROBHD         5 mm PABBO BB-
PULPROG        zg30
TD             65536
SOLVENT        DMSO
NS             8
DS             2
SWH            12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7263477 sec
RG             114
DW             41.600 usec
DE             6.00 usec
TE             294.0 K
D1             1.00000000 sec
TDO            1

-------- CHANNEL f1 --------
NUC1           1H
P1             10.90 usec
PL1            -3.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI             32768
SF             400.1299790 MHz
WDM            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Feb16-2012
EXPO NO                31
PROC NO                1
F2 - Acquisition Parameters
Date_                2012.02.16
Time                21.41
INSTRUM               spect
PROBHD 5 mm PABBO B3-
PULPROG                ZG30
TD                655.36
SOLVENT               DMSO
NS                8
DS                2
SNH                12019.230 Hz
FIDRES       0.183599 Hz
AQ                2.7263477 sec
RG                1.14
DW               41.600 usec
DE                6.00 usec
TE                294.0 K
D1             1.00000000 sec
TD0                1

--------- CHANNEL f1 ---------
NUC1                1H
T1                10.90 usec
PL1                -3.00 dB
SFO1        400.1324710 MHz
F2 - Processing parameters
SI                32768
SF                400.1299790 MHz
WDW                0
SSB                0
LB                0.30 Hz
GB                0
FC                1.00

BRUKER AVANCE II 400 NMR Spectrometer
SAIF
Panjab University Chandigarh

avtar_saifpu@yahoo.co.in
Table: 28 (b)

**Spectral Data**

![Diagram of molecule]

R = n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra δ (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decyloxy</td>
<td>0.872 (–CH$<em>3$ of OC$</em>{10}$H$_{21}$group), 1.265 (Triplet) (–CH$<em>2$)$<em>n$ Polymethylene group of –OC$</em>{10}$H$</em>{21}$), 4.000 (Triplet) (–OCH$<em>2$–CH$<em>2$– of –OC$</em>{10}$H$</em>{21}$), 6.90 &amp; 6.92 (–CH=CH-CO-group), 7.88, 7.91 &amp; 8.07 (p-substituted phenyl ring), The structure confirms by NMR</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>0.873 (–CH$<em>3$ of OC$</em>{12}$H$_{25}$group), 1.258 (–CH$<em>2$)$<em>n$ Polymethylene group of –OC$</em>{12}$H$</em>{25}$), 4.008 (Triplet) (–OCH$<em>2$–CH$<em>2$– of –OC$</em>{12}$H$</em>{25}$), 6.89 &amp; 6.91 (broad) (–CH=CH-CO-group), 7.88, 7.91 &amp; 8.07 (p-sub. benzene ring), The structure confirms by NMR</td>
</tr>
</tbody>
</table>
Table: 29 Texture

![Chemical Structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butyloxy</td>
<td>Threaded Nematic</td>
</tr>
<tr>
<td>Hexadecyloxy</td>
<td>Schlieren Nematic</td>
</tr>
<tr>
<td>Hexyloxy</td>
<td>Threaded Nematic and Smectic-A</td>
</tr>
</tbody>
</table>
2.11 Homologous series-8

Table: 30 Transition temperatures in °C

\[
\begin{array}{cccc}
\text{Sr.No.} & \text{R=} n\text{-Alkyl group} & \text{Smectic} & \text{Nematic} & \text{Isotropic} \\
1 & \text{Methyl} & - & - & 202.0 \\
2 & \text{Ethyl} & - & - & 203.0 \\
3 & \text{Propyl} & - & 168.0 & 184.0 \\
4 & \text{Butyl} & - & 160.0 & 186.0 \\
5 & \text{Pentyl} & - & 140.0 & 188.0 \\
6 & \text{Hexyl} & - & 123.0 & 183.5 \\
7 & \text{Octyl} & - & 108.0 & 165.0 \\
8 & \text{Decyl} & - & 110.0 & 145.0 \\
9 & \text{Dodecyl} & - & 106.0 & 132.0 \\
10 & \text{Tetradecyl} & - & 96.0 & 123.0 \\
11 & \text{Hexadecyl} & - & 100.0 & 118.0 \\
\end{array}
\]

R= n-Alkyl chain
Figure: 16 Phase behavior of homologous series - 8
Table: - 31 Elemental Analysis

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R=n-alkyl chain</th>
<th>Molecular Formula</th>
<th>Calculated %</th>
<th>Observed %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
</tr>
<tr>
<td>1.</td>
<td>Methyl</td>
<td>C_{23}H_{17}O_{6}N</td>
<td>68.49</td>
<td>4.22</td>
</tr>
<tr>
<td>2.</td>
<td>Ethyl</td>
<td>C_{24}H_{19}O_{6}N</td>
<td>69.06</td>
<td>4.56</td>
</tr>
<tr>
<td>3.</td>
<td>Propyl</td>
<td>C_{25}H_{21}O_{6}N</td>
<td>69.60</td>
<td>4.87</td>
</tr>
<tr>
<td>4.</td>
<td>Butyl</td>
<td>C_{26}H_{23}O_{6}N</td>
<td>70.11</td>
<td>5.17</td>
</tr>
<tr>
<td>5.</td>
<td>Pentyl</td>
<td>C_{27}H_{25}O_{6}N</td>
<td>70.59</td>
<td>5.45</td>
</tr>
<tr>
<td>6.</td>
<td>Hexyl</td>
<td>C_{28}H_{27}O_{6}N</td>
<td>71.03</td>
<td>5.71</td>
</tr>
<tr>
<td>7.</td>
<td>Octyl</td>
<td>C_{30}H_{31}O_{6}N</td>
<td>71.86</td>
<td>6.19</td>
</tr>
<tr>
<td>8.</td>
<td>Decyl</td>
<td>C_{32}H_{35}O_{6}N</td>
<td>72.59</td>
<td>6.62</td>
</tr>
<tr>
<td>9.</td>
<td>Dodecyl</td>
<td>C_{34}H_{39}O_{6}N</td>
<td>73.25</td>
<td>7.00</td>
</tr>
<tr>
<td>10.</td>
<td>Tetradecyl</td>
<td>C_{36}H_{43}O_{6}N</td>
<td>73.85</td>
<td>7.35</td>
</tr>
<tr>
<td>11.</td>
<td>Hexadecyl</td>
<td>C_{38}H_{47}O_{6}N</td>
<td>74.39</td>
<td>7.67</td>
</tr>
</tbody>
</table>
Table: 32 (a) IR Spectral Data

![Chemical Structure](image)

R= n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>IR Spectra cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexyloxy</td>
<td>625 (Polymethylene-(CH2)n-group of –OC₆H₁₃)</td>
</tr>
<tr>
<td></td>
<td>1180 (CO of OC₆H₁₃),</td>
</tr>
<tr>
<td></td>
<td>1250, 1600 &amp; 1685 (-COO-group),</td>
</tr>
<tr>
<td></td>
<td>825 (–CH=CH-group),</td>
</tr>
<tr>
<td></td>
<td>1435 (–NO₂ group),</td>
</tr>
<tr>
<td></td>
<td>770 (m-sub. benzene ring),</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by IR.</td>
</tr>
<tr>
<td>Dodecyloxy</td>
<td>645 (polymethylene-(CH₂)n-group of OC₁₂H₂₅)</td>
</tr>
<tr>
<td></td>
<td>1165 (C-O group of -OC₁₂H₂₅),</td>
</tr>
<tr>
<td></td>
<td>1250, 1605 &amp; 1685 (–COO group),</td>
</tr>
<tr>
<td></td>
<td>930 (–CH=CH-),</td>
</tr>
<tr>
<td></td>
<td>1435 (–NO₂),</td>
</tr>
<tr>
<td></td>
<td>820 (p-substituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>770 (m-substituted phenyl ring),</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by IR.</td>
</tr>
</tbody>
</table>
null
BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME          Feb16-2012
EXPNO         41
PROCNO         1

F2 - Acquisition Parameters
Date_          20120216
Time           21.46
INSTRUM        spect
PROBHD         5 mm PABBO BB-
PULPROG        zg30
TD             65536
SOLVENT        DMSO
NS             8
DS             2
SWH            12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7263477 sec
PG             161
DW             41.600 usec
DE             6.00 usec
TE             294.0 K
D1             1.00000000 sec
TD0           1

-------- CHANNEL f1 --------
NUC1           1H
P1             10.90 usec
PL1            -3.00 dB
SF01          400.1324710 MHz

F2 - Processing parameters
ST             32768
SF             400.1299823 MHz
WDW            EM
SSB             0
LB             0.30 Hz
GB             0
PC             1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME: Feb16-2012
EXPNO: 41
PROCNO: 1

F2 - Acquisition Parameters
Date: 20120216
Time: 21.46
INSTRUM: spect
PROBHD: 5 mm PABBO BB-
FUPROG: zq30
TD: 65536
SOLVENT: DMSO
NS: 8
DS: 2
SWH: 12019.230 Hz
FIDRES: 0.183399 Hz
AQ: 2.7263477 sec
RG: 161
DW: 41.600 usec
DE: 6.00 usec
TE: 294.0 K
DL: 1.00000000 sec
TD0: 1

--------- CHANNEL f1 ---------
NUC1: 1H
P1: 10.90 usec
PL1: -3.00 dB
SF01: 400.1324710 MHz

F2 - Processing parameters
SI: 32768
SF: 400.1299823 MHz
WDW: EM
SSB: 0
LB: 0.50 Hz
GB: 0
PC: 1.00

avtar_saifpu@yahoo.co.in
Current Data Parameters
NAME         Feb16-2012
EXPN0                51
PROCNO                1

F2 - Acquisition Parameters
Date_        20120216
Time                21.51
INSTRUM       spect
PROBD            5 mm PABBO BB-
PULPROG   zg30
TD             65536
SOLVENT       DMSO
NS                8
DS                  2
SWH        12019.230 Hz
FIDRES  0.183399 Hz
AQ        2.7263477 sec
RG                322
DW        41.600 usec
DE                 6.00 usec
TE        294.0 K
D1           1.0000000 sec
TDO                1

------- CHANNEL f1 -------
NUCL      1H
F1        10.90 usec
FL1        -3.00 dB
SFOL  400.1324710 MHz

F2 - Processing parameters
SI          32768
SF  400.1299485 MHz
WDW        0M
SSB                 0
LB           0.30 Hz
GB                  0
PC                1.00

avtar_saifpu@yahoo.co.in
Table: 32 (b) NMR Spectral Data

\[ \text{R}= n\text{-Alkyl chain} \]

<table>
<thead>
<tr>
<th>Homologue</th>
<th>NMR Spectra ( \delta ) (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Octyloxy</td>
<td>0.876 ((-\text{CH}_3\text{ of } \text{OC}<em>8\text{H}</em>{17}\text{ group}),)</td>
</tr>
<tr>
<td></td>
<td>1.279 ((\text{Triplet}) (-\text{CH}<em>2-)</em>{n}\text{ Polymethylene group of } \text{OC}<em>8\text{H}</em>{17}\text{),})</td>
</tr>
<tr>
<td></td>
<td>4.006 ((\text{Triplet}) (-\text{OCH}_2\text{-CH}_2-\text{ of } \text{OC}<em>8\text{H}</em>{17}),)</td>
</tr>
<tr>
<td></td>
<td>6.990 ((-\text{CH}=\text{CH}-\text{CO-group}),)</td>
</tr>
<tr>
<td></td>
<td>3.223 ((\text{broad}) (-\text{CH}=\text{CH- group}),)</td>
</tr>
<tr>
<td></td>
<td>6.906 &amp; 6.9278 ((\text{p-sub. benzene ring}),)</td>
</tr>
<tr>
<td></td>
<td>7.885 &amp; 7.907 ((\text{m-sub. benzene ring}),)</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by NMR</td>
</tr>
<tr>
<td>Decyloxy</td>
<td>0.876 ((-\text{CH}<em>3\text{ of } \text{OC}</em>{10}\text{H}_{21}\text{ group}),)</td>
</tr>
<tr>
<td></td>
<td>1.256 ((\text{-CH}<em>2-)</em>{n}\text{ Polymethylene group of } \text{OC}<em>{10}\text{H}</em>{21}\text{),})</td>
</tr>
<tr>
<td></td>
<td>4.013 ((\text{Triplet}) (-\text{OCH}<em>2\text{-CH}<em>2-\text{ of } \text{OC}</em>{10}\text{H}</em>{21}),)</td>
</tr>
<tr>
<td></td>
<td>4.289 &amp; 4.30 ((\text{broad}) (-\text{CH}=\text{CH}-\text{CO-group}),)</td>
</tr>
<tr>
<td></td>
<td>7.764 &amp; 7.925 ((\text{p-sub. benzene ring}),)</td>
</tr>
<tr>
<td></td>
<td>6.903 ((\text{m-sub. benzene ring}),)</td>
</tr>
<tr>
<td></td>
<td>The structure confirms by NMR</td>
</tr>
</tbody>
</table>
Table: 33 Texture

![Chemical structure](image)

R=n-Alkyl chain

<table>
<thead>
<tr>
<th>Homologue</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butyloxy</td>
<td>Threaded nematic</td>
</tr>
<tr>
<td>Hexyloxy</td>
<td>Schlieren nematic</td>
</tr>
</tbody>
</table>