



CHEMISTRY

## SYNTHESIS AND CHARACTERISATION OF BANANA-SHAPED LIQUID CRYSTAL COMPOUND DERIVED FROM ISOPHTHALIC ACID AS A CENTRAL CORE

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### Abstract

The synthesis and mesomorphic properties of a homologous series of schiff's base bent core liquid crystal compounds are reported. Two amino groups are connected to a central part isophthalic acid bis(4-formyl-phenyl)ester. The terminal alkoxy chains have been varied from 4 to 11 and 16 carbon atoms in length. All the compounds exhibit liquid crystalline phases which were stable in nature. Only the A-16 compound show a show nematic phase along with B1 phase. All the synthesized nine compounds exhibited enantiotropic liquid crystal phase. The transition temperatures and phase behaviours were studied by Differential Scanning Calorimetry (DSC), Polarizing Optical Microscopy (POM).

**Keywords:** Liquid Crystal, Schiff's base, Isophthalic acid, Banana-shaped compounds

### Introduction

It has been well documented that the study of liquid crystalline compounds is very important for the continual development and understanding of the field of molecular engineering. It has been an active and fruitful research area for many years and academic and application – based interests have found a place within materials science. Several types of liquid crystals have been discovered; today banana shaped liquid crystals occupy one of the most exciting areas of research in both mesogenic materials and supramolecular chemistry. It has been the subject of extensive investigations in the last years because they can form new mesophases with unusual properties which are different from those of calamitic mesogens. Due to their intrinsic shape the molecules can be packed in a polar fashion which leads to a long-range correlation of the lateral dipoles. The origin of this novel area stems from the use of bent shaped molecules, which adopt a compact packing arrangement that restricts rotational freedom, thus allowing the molecules to organize in to novel types of liquid crystalline phases. In the past several years great number of banana shape compounds have been synthesized and characterized [1-4] and at least eight different phases (chronologically assigned as B<sub>1</sub>.....B<sub>8</sub>) have been described.[5,6,7,8].Among them the electro-optical switching has been observed only in the smectic B<sub>2</sub>, B<sub>5</sub> and B<sub>7</sub> phases. The most widely studied B<sub>2</sub> phase is identified as tilted antiferroelectric polar smectic (SmCP<sub>A</sub>) phase with either synclinic (SmC<sub>s</sub>PA) or

anticlinic (SmC<sub>a</sub>PA) structures [9]. B<sub>7</sub> phase is very unique and interesting due to its beautiful and extra ordinary textures, and it may have a double tilted director structure with C1 symmetry [10].The B<sub>3</sub> and B<sub>4</sub> phases are considered to be crystalline phases. Recently a growing number of bent-core materials have been found to show nematic phases, too [11].The B1 phase has been designated as a columnar phase with rectangular lattice. It is found that banana mesophases are very sensitive to the substituents connected to different positions of the aromatic rings keeping constant the molecular architecture. The situation is more complicated if the linking groups are changed for vinyl carboxylate or ester groups. More and more banana shaped compounds with ester connecting groups were published [12].Systematic work was carried out on investigating the influence of the carboxyl function on the mesomorphic properties in five aromatic ring bananas building up symmetrical and unsymmetrical structures[13].Schiff's base type compounds containing seven aromatic rings with ester connecting groups have been studied too. These compounds show B1 or B2 meosphases [14].

The relationship between the molecular structures and their mesomorphic properties is one of the most intensively studied topics for banana shaped liquid crystals. Many aspects concerning this banana shaped liquid crystal compounds including the mesophases exhibited by them, the phase structures and some of the physical properties have been reviewed [1, 15-18]. For instance, banana shaped compounds with different central cores, with different linkages in the wings of the

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molecules and with varying lengths of the terminal alkyl tails have been extensively studied. The linking groups between the wings of the aromatic fragments of the bent shaped molecule can also be varied, and the most common are the imine and ester linking groups. The length of the terminal alkyl (oxy) chain also has a pronounced effect on the mesomorphic properties of compounds with bent-molecules. In a great amount of material families, with the same core and different terminal chains, it has been found that on increasing the chain length the phase sequence B6-B1-B2 takes place. [19-21]. The B2 and B6 phases are single-layer and intercalated smectics, respectively, whereas the B1 phase is a smectic- frustrated intermediate structure with a two dimensional modulation. To understand the relationship between molecular structure and mesomorphic properties, banana-shaped compounds can be designed in different ways. Most molecules that exhibit banana-phases are composed of symmetrical resorcinol derivatives substituted at the 1 and 3 position [21, 22-29]. Pelzyl et al G.Pelzl, S.Diele, W.Weissflog reported banana-shaped compounds with 2, 7-dihydroxy naphthalene showed B1 phase. In this paper we report the synthesis and mesomorphic properties of the homologues series of compounds derived from isophthalic acid which shows only B1 phase. These compounds are symmetrical five ring esters with the varying terminal alkoxy chain length from 4-11 and 16. The characterization of the mesophases has been carried out using polarizing optical microscopy and differential scanning calorimetry.

## Experimental

### General information

The chemicals procured were used without any further purification. Dichloromethane and acetone, were obtained were purified and dried as per the standard procedures. The target banana compounds were purified by recrystallization choosing appropriate mixture of solvents; while the intermediates were purified by column chromatographic technique.

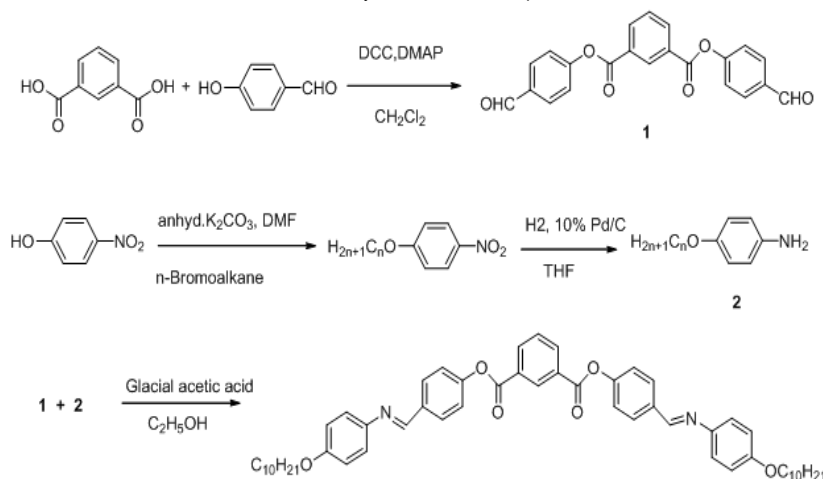
Chromatography was performed using either silica gel (100-200,230-400 mesh). Thin layer chromatography (TLC) was performed on aluminium sheets precoated with silica gel (merck, kieselgel, 60F254). Infra red spectra were recorded on a Perkin-Elmer Spectrum 1000 FTIR spectrometer; the spectral positions (absorption maxima) are given in wave numbers ( $\text{cm}^{-1}$ ). NMR spectra were recorded using Bruker AMX 400(400 MHz) spectrometer. For  $^1\text{H}$  NMR spectra, the chemical shifts are reported in parts per million relative to SiMe<sub>4</sub> (TMS) as an internal standard and coupling constants are presented in Hertz. Elemental analysis was performed using a Eurovector model EA 3000 CHNS elemental analyzer. The identification of mesophases and determination of phase transition temperatures were carried out using a polarizing optical microscope (POM) (Leica DMLP) equipped with programmable hot stage (Mettler FP90).

The target molecule was synthesized in two steps. Firstly, DCC coupling of isophthalic acid and 4-hydroxy benzaldehyde provide the intermediate (a) as white solid. Secondly, O-alkylation of 4-nitro phenol followed by hydrogenation to obtain the intermediate (b). Acid catalyzed condensation of (a) with (b) yielded the final compound as yellow solid.

### Isophthalic acid bis(4-formyl-phenyl)ester (1a)

Isophthalic acid (1g, 0.006 mol), 4-hydroxy benzaldehyde (1.46g, 0.012 mol) and dicyclohexylcarbodiimide (2.68 g, 0.013 mol) were dissolved in dichloromethane. To this a pinch of 4-(N, N-dimethyl)aminopyridine was added and the reaction mixture was stirred at room temperature for 24 h. The white colored compound was removed by filtration and the solvent was evaporated. Recrystallization of crude product from ethanol gave the pure product (1). Yield: 75%: a white solid, melting point: 135-136°C. IR (KBr pellet): 1985, 1769, 1625, 1192, 875  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400MHz)  $\delta$ : 9.8 (s, 1H, 1XCHO), 9.2 (s, 1H, Ar), 8.5 (d, 2H, Ar), 7.6 (d, 1H, Ar), 7.4 (d, 1H, Ar), 7.0 (d, 2H, Ar).

Scheme 1: Synthesis of A-n compounds



4-alkoxy anilines were synthesized by the O-alkylation of 4-nitro phenol followed by hydrogenation with H<sub>2</sub> gas in the presence of palladium on activated carbon (2).

#### Isophthalic acid bis-{4-[(4-decyloxy-phenylamino)-methyl]-phenyl} ester

Condensation of the intermediate (1) and (2) provided the target banana molecule. The product was purified by chromatography on silica gel, and recrystallised several times from dichloromethane /ethanol.

#### Conclusion

In conclusion, we have studied the mesophase behaviour of a series of banana-shaped mesogens, the Isophthalic acid bis-{4-[(4-decyloxy-phenylamino)-methyl]-phenyl} ester. The effect of the length of the alkoxy chains on the mesomorphic behaviour was examined by preparing 9 compounds in which the carbon number of the alkoxy chains is varied from 2 to 11, and 16. Almost all compounds show B1 phase except A-9 because it shows B1 along with nematic phase. Thus the mesomorphic behaviour of the present series of compounds is reminiscent of that seen for

other banana-shaped mesogens reported in the literature.

#### Results and discussion

The identification of the mesophases and determination of phase transition temperatures were achieved with the help of polarizing optical microscope (POM). The sample placed between a clean untreated glass slide and a cover slip was used for this particular study. The mesophase assignment was based on the observation of birefringence and fluidity when examined under POM. To confirm the transition temperatures and to determine their associated enthalpies, a differential scanning calorimeter (DSC) calibrated using pure indium was employed. The peaks observed in DSC traces (obtained at a rate of 10°C/min.) due to phase transition was reproducible and peak temperatures were consistent with those deduced from the optical experiments.

A total of nine symmetric bent core molecules were synthesized. In an effort to understand the structure-property relations the terminal alkoxy chain lengths have been varied. The transition temperatures and enthalpy values for the bent core molecules are summarized in table 1. As can be seen the entire nine compounds exhibit an enantiotropic phase and the textures seen are different for all the compounds

**Table 1:** Phase transitions temperatures (°C) and the corresponding enthalpies (J/g) of liquid crystalline banana compound

| Compound | n  | Cr1 | Heating<br>Cooling         | Cr2 | Heating<br>Cooling           | B1 | Heating<br>Cooling        | N | Heating<br>Cooling        | I |
|----------|----|-----|----------------------------|-----|------------------------------|----|---------------------------|---|---------------------------|---|
| A4       | 4  | •   | 138.7[29.2]<br>121.2[13.3] | -   | -                            | •  | 186.9[5.9]<br>185.5[1.6]  | - | -                         | • |
| A6       | 6  | •   | 150.2[46.4]<br>161.3[52.4] | -   | -                            | •  | 179.9[1.0]<br>169.3[9.1]  | - | -                         | • |
| A7       | 7  | •   | 159.9[61.0]<br>70.9[44.7]  | -   | -                            | •  | 183.9[16.5]<br>182.3[0.6] | - | -                         | • |
| A8       | 8  | •   | 125.6[51.9]<br>132.8[27.8] | -   | -                            | •  | 182.5[8.1]<br>182.4[0.8]  | - | -                         | • |
| A9       | 9  | •   | 150.7[27.0]<br>68.5[13.4]  | -   | -                            | •  | 175.4[1.1]<br>172.7[14.9] | • | 186.5[1.0]<br>184.1[0.51] | • |
| A10      | 10 | •   | 131.1[19.1]<br>119.1[31.5] | •   | 164.7[19.0]<br>166.6[26.1]   | •  | 183.7[1.1]<br>187.2[7.2]  | - | -                         | • |
| A11      | 11 | •   | 81.3[18.9]<br>5.3[19.3]    | •   | 147.6[24.0]<br>143.8[18.6]   | •  | 174.2[13.7]<br>171.6[1.2] | - | -                         | • |
| A16      | 16 | •   | 93.3[47.0]<br>78.3[39.0]   | •   | 130.27[22.5]<br>120.03[12.3] | •  | 142.5[0.75]<br>139.4[2.1] | - | -                         | • |

In compound A-16, there are endothermic peaks at 93.3, 130.2 and 142.5°C on heating indicate the existence crystal to crystal transition and of B1 phase. On cooling, crystal to crystal transition and the B1 phase appears from isotropic phase to the super cooled region. It is already reported that the introduction of a central substituent in the 1, 3 phenylene moiety is decrease the smectic thermal

stability [27, 28]. In the present study the thermal stability of B1 phase is decreased without any substituent in the Schiff's base moiety. When the isotropic liquid is cooled slowly, complicated optical textures of B1 phase is observed and the texture variants mostly occur simultaneously within the same preparation. Fig.1 form of B1 phase of A-11 at 143.8°C.

Plastic phase was observed for the compound having terminal alkoxy chain length five i.e A-5.

Fig.1. Polarising optical microscope texture of B1 phase of A-11 at 143°C



#### Compound A-4

Yield: 45%: a yellow solid [found C, 75.37; H, 5.97; N, 4.23.  $C_{42}H_{40}N_2O_6$  requires C, 75.43; H, 6.03; N, 4.19]. IR (KBr pellet): 2928, 1755, 1664, 1609, 1267, 836, 717  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.02 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.44 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.90 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.95-1.43 (m, 4H, 2XCH<sub>2</sub>), 0.91-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-6

Yield: 50%: a yellow solid [found C, 76.27; H, 6.59; N, 3.79.  $C_{46}H_{48}N_2O_6$  requires C, 76.22; H, 6.67; N, 3.86]. IR (KBr pellet): 2928, 1752, 1664, 1609, 1267, 839, 717  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.01 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.43 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.91 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.95-1.42 (m, 8H, 4XCH<sub>2</sub>), 0.91-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-7

Yield: 52%: a yellow solid [found C, 76.62; H, 7.02; N, 3.81.  $C_{48}H_{52}N_2O_6$  requires C, 76.57; H, 6.96; N, 3.72]. IR (KBr pellet): 2928, 1751, 1664, 1609, 1269, 839, 717  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.02 (d, J=9 Hz, 1H, Ar), 8.48 (s, 1H, 1XCH=N), 8.49 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.43 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.91 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.95-1.42 (m, 10H, 5XCH<sub>2</sub>), 0.90-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-8

Yield: 52%: a yellow solid [found C, 76.94; H, 7.32; N, 3.51.  $C_{50}H_{56}N_2O_6$  requires C, 76.89; H, 7.23; N, 3.59]. IR (KBr pellet): 2927, 1751, 1664, 1609, 1269, 836, 717  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.02 (d,

J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.47 (m, 2H, Ar), 8.09 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.43 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.91 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.94-1.42 (m, 12H, 6XCH<sub>2</sub>), 0.89-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-9

Yield: 57%: a yellow solid [found C, 77.14; H, 7.54; N, 3.55.  $C_{52}H_{60}N_2O_6$  requires C, 77.20; H, 7.48; N, 3.46]. IR (KBr pellet): 2928, 1751, 1664, 1619, 1269, 836, 715  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.02 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.43 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.90 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.94-1.42 (m, 14H, 7XCH<sub>2</sub>), 0.89-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-10

Yield: 59%: a yellow solid [found C, 77.53; H, 7.77; N, 3.42.  $C_{54}H_{64}N_2O_6$  requires C, 77.48; H, 7.71; N, 3.35]. IR (KBr pellet): 2918, 1751, 1664, 1613, 1269, 836, 714  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.02 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.09 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.44 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.90 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.94-1.42 (m, 16H, 8XCH<sub>2</sub>), 0.89-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-11

Yield: 63%: a yellow solid [found C, 77.68; H, 7.83; N, 3.18.  $C_{56}H_{68}N_2O_6$  requires C, 77.74; H, 6.36; N, 4.02]. IR (KBr pellet): 2918, 1749, 1664, 1613, 1254, 836, 714  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.03 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.44 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.90 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.94-1.42 (m, 18H, 9XCH<sub>2</sub>), 0.89-0.86 (t, 3H, 1XCH<sub>3</sub>).

#### Compound A-16

Yield: 65%: a yellow solid [found C, 78.85; H, 8.76; N, 2.87.  $C_{66}H_{88}N_2O_6$  requires C, 78.84; H, 8.82; N, 2.79]. IR (KBr pellet): 2918, 1746, 1660, 1603, 1254, 836, 714  $cm^{-1}$ .  $^1H$  NMR ( $CDCl_3$ , 400MHz)  $\delta$ : 9.03 (d, J=9 Hz, 1H, Ar), 8.49 (s, 1H, 1XCH=N), 8.48 (m, 2H, Ar), 8.08 (d, J=6.8 Hz, 2H, Ar), 7.74 (m, 1H, Ar), 7.45 (d, J=6.8Hz, 2H, Ar), 7.37 (d, J=8.8 Hz, 2H, Ar), 6.93 (d, J=8.4 Hz, 2H, Ar), 3.98-3.90 (t, J=6.8Hz, 1XOCH<sub>2</sub>), 1.94-1.41 (m, 28H, 14XCH<sub>2</sub>), 0.89-0.86 (t, 3H, 1XCH<sub>3</sub>).

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