# EXPERIMENTAL STUDIES ON SOME LIQUID CRYSTALLINE COMPOUNDS EXHIBITING SMECTIC ORDER

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for the Degree of
Doctor of Philosophy

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#### **DECLARATION**

I hereby declare that the thesis was composed by me independently and that it has not formed the basis for the award of any Degree, Diploma, Associateship, Fellowship or other similar title.

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I certify that this thesis has been composed by Sri B.S.Srikanta based on investigations carried out by him at the Liquid Crystals Laboratory, Raman Research Institute, under my supervision. The subject matter of this thesis has not previously formed the, basis of the award of any degree, diploma, associateship, fellowship or other similar title.

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#### PREPACE

This thesis describes some experimental investigations carried out by the author on the nematic and smeetic phases exhibited by several compounds and mixtures. The studies may be broadly classified as follows.

- The first quantitative study of the effect
  of a curl n distortion on the smectic A nematic
  transition point which is analogous to the
  influence of a magnetic field on the superconductor-normal metal transition (chapter II).
- studies on the physical properties of several structurally related new mesogenic compounds. Interestingly, these investigations have led to the observation of certain unusual properties like large thermal contraction of the bilayer spacing, S<sub>A</sub>-S<sub>A</sub> transition and the reversal of the sign of dielectric anisotropy (chapters III. IV and V).
- Investigations on two binary mixtures exhibiting an induced smeetic A phase, the remarkable feature of these mixtures being that they show in addition to the commonly observed maximum, a well defined minimum in the smeetic A-nematic transition boundary (chapter VI).

4 Effect of skewed cybotactic short range order on the dielectric and conductivity properties of binary mixtures exhibiting the nematic phase. A striking new result obtained la these studies is that the dielectric anisotropy of a mixture reverses its sign, unusually becoming negative at higher temperatures (chapter VII).

In chapter I. We give a general introduction to the subject matter of the thesis and describe the background work on the topics discussed la later chapters. The salient new results contained in chapters II - VII are summarized below.

The order parameter characterizing the emectic A phase has two components, viz., the amplitude and the phase of the one dimensional density wave. This is analogous to the order parameter of a superfluid.

Purther, constancy of the layer thickness in the smectic A phase does not allow a curl n type of distortion, where n is a unit vector, called the director, along the preferred direction of orientation of the molecules. This is analogous to the expulsion of a magnetic field by a superconductor. Recognising this analogy between the second order smectic A-nematic transition under curl n distortion and superconjuctor-

the phase diagram of Tan as a function of twist deformation. In the first, the twist deformation is produced by applying a magnetic field to a homogeneously aligned sample. The transition point was detected by monitoring the light scattered by bend fluctuations in the sample. However, as will be discussed in chapter II, this technique does not yield quantitative results. In the second method, we used a wedge shaped twisted negational to get the quantitative data on the reduction of

Tan of N-p-cyanobenzylidene-p'-octyloxyaniline (CBOCA)(fig.1) which has almost second order A-N transition, and 4-n-octyloxy-4'-cyanobiphenyl (8 OCB) which has a very weak first order A-N transition, as a function of twist deformation.

strongly polar compounds have led to two important discoveries, viz., reentrance and smectic A polymorphism. These phenomena result from the subtle changes taking place in the bilayer structures of such compounds. The bilayer structure depends upon the mutual disposition of various dipolar groups in the molecules. Notivated by these results, we undertook experimental investigations on various physical properties of several structurally related liquid crystalline compounds most of them possessing strongly polar end groups with bulky lateral methyl or methoxy substituents.

In chapter III, we describe Xray investigations on the thermal evolution of layer spacings in the smectic A phases of 4-n-alkylphenyl-3'-methyl-4'-(4"-cyanobenzoyloxy)benzoates (nPMCBB)(see fig.2 for structural formula), their nitro analogues (nPMBB), nPMeOCBB (obtained by replacing lateral methyl group

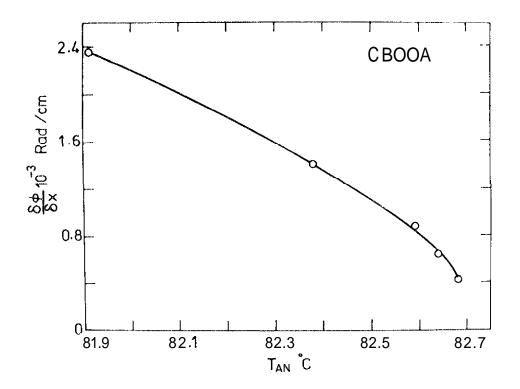


FIGURE 1

Variation of  $T_{AN}$  with  $\delta \phi/\delta x$  for CBOOA.

of nPMCBB by the bulkier methoxy group), nCPMeCBB (obtained by reversing cyano and alkyl end groups in nPMeOOBB). nPMeOBrBB (obtained by replacing the cyano end group of nPMeCCBB with a bromine atom) and 4-biphenylyl-4"-n-undecyloxybenzoate (BO11). Our studies indicate several striking differences: in the thermal variation of layer spacings amongst these compounds. nPMOBB, nPMMBB and nPMeOOBB compounds in which the dipole moments of the linkage groups oppose that of the terminal polar cyano (or nitro) group. exhibit large bilayer spacings (>1.61. 1 is the molecular length) which are extremely sensitive to temperature. In all these cases, the lager spacing increases enormously as the temperature is lowered in (fig.3)the smectic A phase. More interestingly, 10 PMOBB exhibits a small jump in the layer spacing (fig. 4) at  $T_{an} - T \simeq 15^{\circ}$ . This corresponds to an  $A_d - A_2$ transition according to the nomenclature introduced by the Bordeaux group. 9 nCPMeOBB compounds, in which the dipole moments of the ester groups are aligned parallel to that of the terminal cyano group, have smaller bilayer spacings (21.3人) and show very weak thermal dependence. These results are discussed in

of a modal in which mutual interactions astween a pair of molecules is considered (fig.5). It can be

4 - cyanophenyl -- 3' -- methyl -- 4'-(4"-n -- alkyl benzoyloxy) benzoates. (n C PMBB)

$$NC \longrightarrow C \longrightarrow C \longrightarrow C \longrightarrow C \cap H_{2n+1}$$

4-n—alkylphenyl—3—methyl—4-(4—cyanobenzoyloxy) benzoates

#### (nPMCBB)

4-n-atkylphenyl -3- methyl -4-(4-ndrobenzoyloxy) benzoates

#### (n PMNBB)

4-n-alkylphenyl-3-methoxy-4-(4-bromobenzoyloxy) benzoates
(nPMeOBrBB)

$$NC - C_n H_{2n+1}$$

4-n-alkylphenyl-3-methoxy-4-(4-cyanobenzoyloxy) benzoates (nPMeoCBB)

4 -cyanophenyl -3'-methoxy -4'-(4''-n -alkyl benzoyloxy) benzoates (n CPMeOBB)

4-biphenylyl -4''-n - undecyloxy benzoate (B011)

# FIGURE 2: Structural formulae and acronyms of the compounds studied

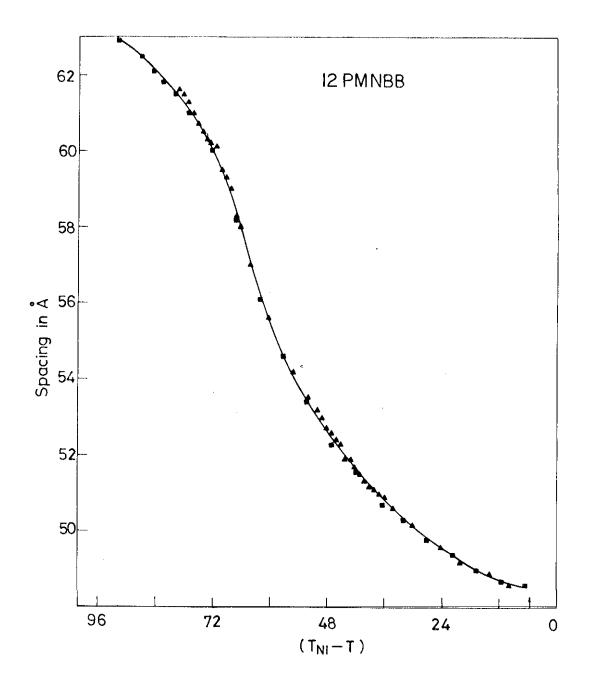
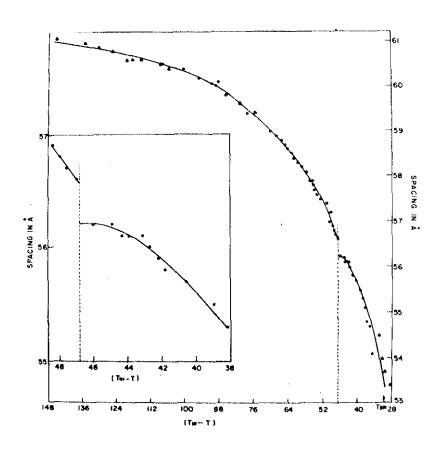


FIG.3: Temperature variation of the layer spacing of 12 PMNBB. ( $T_{NI}$  T) is the relative temperature,  $T_{NI}$  being the nematic-isotropic transition point. The arrow mark indicates  $T_{AN}$ .



### FIGURE 4

Temperature variation of the layer spacing of 10 PMCBB. •, and 4 represent measurements accurate to ±0.1 Å and ±0.3 Å respectively. An A<sub>d</sub>-A<sub>2</sub> transition occurs with a jump in the layer spacing at the temperature corresponding t~ the dashed line. The region around this transition point is shown on a magnified scale in the inset, The arrow mark indicates the A-N transition point.

seen that neighbouring nCPMBB (or nCPMcOBB) molecules possess minimum interaction energy when their aromatic cores overlap (fig.5a). On the other hand, the dipolar interaction between the polar end group of one molecule and an ester dipole 03 a neighbouring molecule (fig. 5b) in nPMCBB, nPMRBB and nPMeOCBB series is repulsive. and the interaction energy can be expected to be lower when the overlapping takes place near the polar ends (fig. 5c). This leads to the differences in the bilayer spacings and their thermal dependences shown by the two types of molecules. Further, in nPMCBB and similar series of compounds, the interaction is confined to one end of the molecule and the structure is rather fragile. As the temperature is raised, the structure with overlapping near the polar ends breaks up tends to take up the configuration with overlapping of the aromatic cores. This lo a consequence of the relatively weak intermolecular interactions fa these x because of the presence of bulky lateral e methyl or methoxy group. As a result, the layer spacing decreases with increase of temperature. nPMeOBrBB compounds exhibit monolayer smectics. Unlike compounds without any lateral substituents. 10.11 nPMeOBrBB compounds exhibit considerable thermal

## FIGURE 5

Schematic diagrams showing the disposition of various dipolar groups of a pair of (a) nCPMBB, (b) nPMCBB molecules with an overlap of the aromatic cores, and (c) a pair of nPMCBB molecules with an overlap of the polar end groupe.

expansion of the layer spacing in the smeetic A phase again due to the weaker intermolecular interactions because of the lateral methoxy group.

As we have seen in the previous paragraph. some of the compounds mentioned earlier show large thermal contraction of the bilayer spacings while others show weak thermal dependence. In order to see the effect of such changes in the bilayer structure on the dielectric properties, we have also undertaken dielectric studies (both static and dispersion studies up to 13 MHs) on some of them. The results are presented in chapter IV. The dielectric properties of the compounds are in conformity with the model described in the previous paragraph. In some at the nPMNBB and nPMCBB compounds, the dielectric anisotropy ( $\triangle \epsilon = \epsilon_{..} - \epsilon_{.}$ ) reverses sign becoming negative at lower temperatures in the smectic A phase (fig.6). Such a reversal can be expected even an the basis of the dielectric theory of nematice due to Maier and Meier 12 if the net dipole moment of the molecule makes an appropriate angle with the long axis. In nPMCBB and nPMNBB compounds, there is an additional contribution to the reversal of As from the variation of the bilayer structure. As we have seen earlier, with

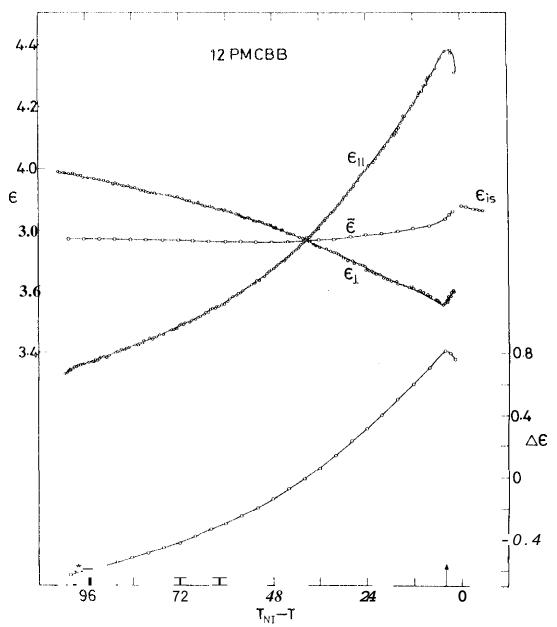
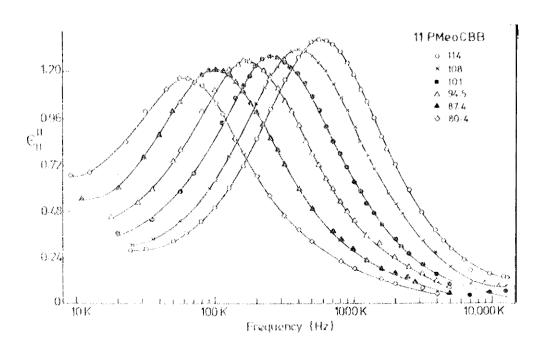


FIG.6: Temperature variatione of the low frequency dielectric Constants (upper part) and the dielectric anisotropy (lower part) of 12 PMCBB measured at 1592 Hz. T<sub>NI</sub> T is the relative temperature, T<sub>NI</sub> being the N-I transition temperature. The arrow mark indicates T<sub>AN</sub>.

lowering of temperature, a large number of molecules associate in pairs with an overlapping near the polar ends as in fig.5c, thus giving rise to an increasing compensation of the longitudinal dipole moments. This obviously leads to a lowering of  $\Delta \epsilon$  at lower temperatures.

We also present in Chapter IV, the dielectric dispersion studies on some of the above mentioned compounds. The differences in the bilayer structure between nPMeOCBB and nCPMeOBB are reflected in the relaxation process also. For instance, in 11 PMeOCDB in which the dipole moments of the ester groups oppose that of the terminal cyano group, the si peak & --with increase of temperature (fig.7) though one ~ @ ~ 1 a normally expect a progressive reduction in this level as the temperature is increased. This is again a consequence of the fragility of the bilayer structure of the compound and the consequent enhancement in the effective dipole moment per molecule at higher temperatures of the smectio A phase. Even in 11 OFMeOBB and 12 CPMeODB, in which the dipole moments of the ester groups are aligned parallel to that of the terminal cyano group, the pairs as shown in fig.5a break up at higher temperatures due to weak intermolecular interactions,

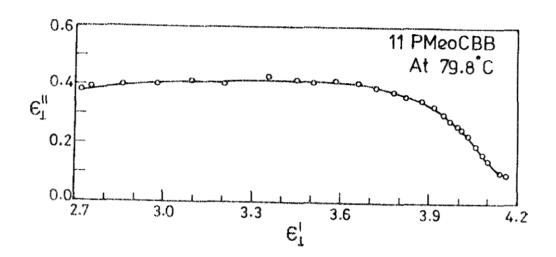


## FIGURE 7

Frequency dependence of all at various temperature a (shown against the symbols) in the smectic A phase of 11 PMeOCBB.

and the peak of t', has practically the same level at all temperatures. Interestingly, the pair formation of 1: PheodBB molecules with an overlapping near the polar ends results in a broad relaxation of 1, with a broad maximum at a particularly low frequency of  $\sim 6$  MHz for this relaxation (fig.8). Apparently in such a structure, the pair reorients as a single unit around the long exis.

Usually in the nematic phase, the electrical conductivity which arises due to ionic impurities, is greater in a direction parallel to the director  $(\sigma_{ii})$  than that perpendicular to the director On the other hand,  $a_i < \sigma_{ii}$  in the smectic A phase as the ions can flow more easily in the plane of the smectic layers than in a direction perpendicular to them. Larlier studies have clearly shown that the conductivity emisotropy is strongly influenced by the short range order in the medium. In particular, studies by Mirosa-Roussel et al. have demonstrated & hat the transport properties behave differently is the bilayer



# FIGURE 8

Cole-Cole plat .for  $\epsilon_{\perp}$  relaxation in the case of 11 PMeOCBB

and monolayer smectics. Since there are large variations in the bilayer structures in the smectic A phase exhibited by the compounds mentioned earlier, we undertook measurements of the principal conductivities on these compounds. The results are presented in chapter V.

In most of the compounds studied by us, the conductivity anisotropy ( $\triangle \sigma = \sigma_{11} - \sigma_{2}$ ) changes sign becoming negative at a 'cross over' temperature Tor as the temperature is lowered towards Tam Tor-Tam. the cross over temperature relative to  $T_{AN}$ , increases in the order: 11 PMeOCBB. 11 CPMeOBB and 11 PMeOBrBB. This trend is the same as that of  $\triangle H_{N,s}$ , the heat of AN transition of these compounds. One would usually expect that if  $\triangle H_{NA}$  is small, the smectic like short range order builds up far above Tan and hence the oross over should occur in the nematic phase St a higher temperature relative to TAN. We have interpreted the reverse trend in terms of the permeation process due to layering in the smectic A phase, which results in a lowering of a... The ionic mobility due to the permeation process is  $\propto d^2$ , where d is the layer thickness, which accounts for the observed trend.

In chapter VI, we present studies on the

temperature variations of the layer spacings, dielectric properties (both 'static' and dispersion studies up to 13 MHs) and conductivities bar several compositions of two binary systems, each with a strongly polar component exhibiting a partial bilayer (Ad) smeetic A phase and a weakly polar nematogen. Both the systems exhibit a maximum (indicating induced smeetic A phase) and a we31 defined minimum in the A-N transition boundary. The maximum occurs for approximately 50mole per cent of the components and the minimum occurs for a composition rich in the highly polar component.

The two systems investigated are: (i) p'-nitro phenyl-p-n-octyloxybenzoate (NFOCB) with (2-hydroxy)-p-ethoxybenzylidene-p'-hutylaniline (OH-EBBA) (see fig. 5 for phase diagram), and (ii) p'-n-octyloxy-p-cyanobiphenyl (8 OCB) with p-butoxyphenyl-(p'-pentyloxy)-benzoate (40.05). The minimum in the phase boundary indicates that the partial bilayer of the polar compound is structurally incompatible with the monolayer characteristic of the induced A phase. DSC studies show that the smectic A-nematia transition is strongly first order for mixtures lying near the maximum of the A-N transition boundary while St is almost of second order

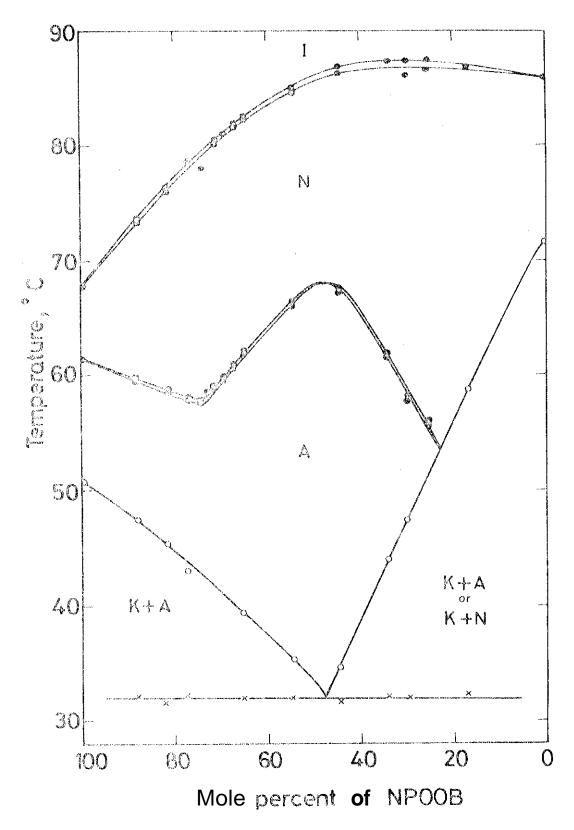


FIG.9: Phase diagram of mixtures of NPOOB with OH-EBBA. K Stands for crystalline phase.

character for compositions lying near the minimum. Due to the presence of the lateral hydroxyl group in OH-EBBA which increases the intermolecular separations, the molecular interactions responsible for the induced smectic A phase are such weaker in system I than in system II. In system I, there is a considerable thermal expansion of the layer spacing, the expansion coefficient attaining a maximum value for the composition corresponding Bo the minimum in the 4-H boundary (fig. 10). But such a trend is not very conspicuous in system II. We attribute this difference to the different strengths of the intermolecular interactions in the two systems. It appears well established that charge transfer complex formation between the strongly polar and weakly polar molecules plays an important part in giving rise to the induced emectic A phase. 17 The competition between the two types of interactions, vis., antiparallel associations between the molecules of strongly polar compounds 18 and charge transfer interactions between the molecules of strongly polar and weakly polar molecules determines the physical properties of the mixtures. The association between the strongly polar and weakly polar molecules which becomes more effective at lower temperatures leads to several interesting effects on the disloctric properties of the mixtures: (1) the strongly polar

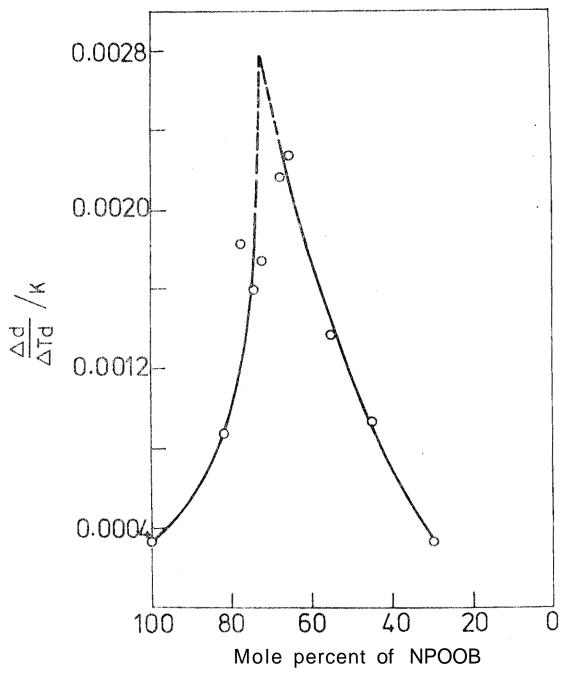


FIG.10: The thermal expansion coefficients of the layer spacings of mixtures of NPOOB with OH-EBBA at  $T_{AN} - T = 2.5^{\circ}$ .

molecules contribute more effectively to the polarization in the mixtures than in the pure compounds in which antiparallel interactions between neighbouring molecules reduce the effective dipole moment contributing to the polarization. Consequently the c. values of the mixtures are larger than the values calculated from an additive law: (2) the charge transfer complexes can be expected to have a considerable transverse polarizability. This results in an increase of a as the temperature is lowered in all the mixtures: (3) the dipolar correlation factors in the layered A phase can be expected to lower su as is clearly seen for compositions close to the first order A transitions near the maximum of the phase boundary. E. decreases with a jump at TAN in these compositions, but as the temperature is lowered in the smectic A phase, the effect (1) mentioned above leads to an increase of  $\varepsilon$ . and hence that of As; (4) on the other hand, for compositions close | o the minimum in the A-N boundary, which have a nearly second order A-N transition, the smectic order and hence the dipolar correlations can be expected to increase as the temperature is lowered. e., hence does not increase much at lower temperatures and effect (2) mentioned above leads to a decrease of As at lower temperatures (fig. 11).

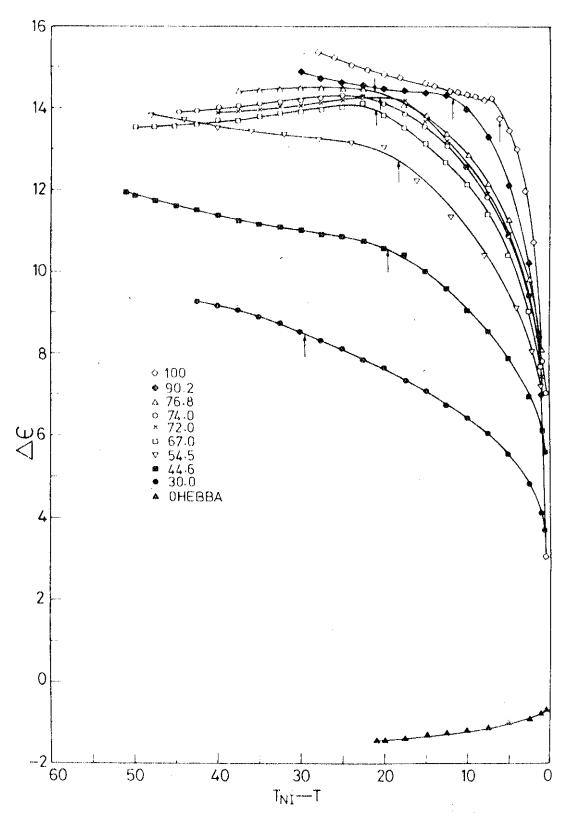


FIG.11: Dielectric anisotropies of mixtures of NPOOB with OH-EBBA as functions of relative temperature. The numbers against the symbols indicate mole percentages of NPOOB and the arrows indicate  $T_{AN}$ .

Further, these interactions also lead to higher activation energy for the relaxation of e., in the mixtures compared to that in the pure highly polar component. In system I, the composition corresponding to the minimum in the A-M boundary has the <u>lowest</u> activation energy in the nematic phase, probably as a consequence of the second order nature of the transition in this case. The smectic like short range order would be quite strong in the nematic phase and this In turn would lead to a lowering of Ma activation energy. In system II, in which measurements can be made over a wide range of temperatures in the induced A phase for compositions close to the maximum of the AN boundary, the activation energy is found to increase continuously as the temperature is lowered (fig. 12).

The conductivity enisotropy changes sign, becoming negative as we cool the sample towards  $T_{AN}$  in all cases. Close to the minimum of the AN boundary of system I.  $(T_{Cr} - T_{AN})$  exhibits a maximum (fig. 15) showing the strong influence of the strength of the AN transition on the transport properties in these systems.

In chapter VII, we study the effect of skewed cybotactic structure on the dislectric constants and conductivities of some binary mixtures exhibiting the

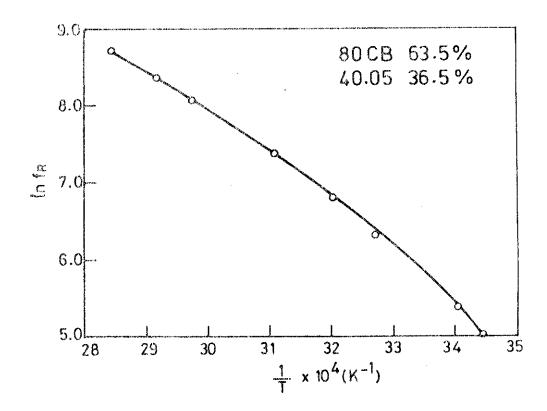


FIG.12: Plat of in  $f_R$  vs.1/T in the case of a mixture of 63.5 mole per cent of 8 0CB with 36.5 mole per cent of 40.05, where  $f_R$  is the relaxation frequency corresponding to  $\epsilon_R^{\rm st}$  peak.

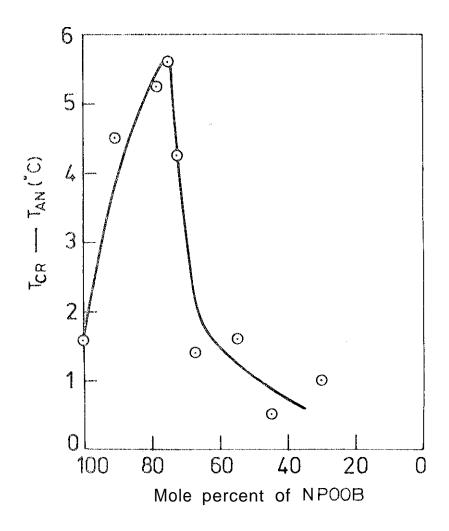


FIGURE 13

Composition-dependence of the relative cross wer temperatures of the conductivities of mixtures of NPOOB with OH-EBBA.

nematic phase. 2-cyano-4-heptylphenyl-4'-pentyl-4-biphenyl carboxylate [7P(2CN)5BC], a nematogen having a cyano group making a large angle (~60°) with the long axis of the molecule, has a skewed cybotactic structure (smectic C like short range order) in the nematic phase. 19 Interestingly, no smectic C phase is observed at lower temperatures. The tilt angle of the skewed cybotactic structure decreases from ~48° at 25°C to ~40° at 87°C. We have studied mixtures of 7P(2CN)5BC with heptyl-cyanobiphenyl (7CB) which has a cyano group along the long axis of the molecule and hence a large positive  $\Delta c$ .

dielectric anisotropy, with the mixtures having values between those of the two pure components. In particular, a mixture of 85 mole per cent of 7P(2CN)5BC with 15 mole per cent of 7CB exhibits very low values of \$\triangle c\$, and further, a reversal of sign of \$\triangle c\$ as the temperature is varied, unusually becoming negative at higher temperatures (fig.14). Such a trend has been observed for the first time. This trend is opposite to that predicted by the Maier-Meier theory of the dielectric properties of nematics. The reverse trend can be accounted for on the basis of the temperature variation of the tilt angle of

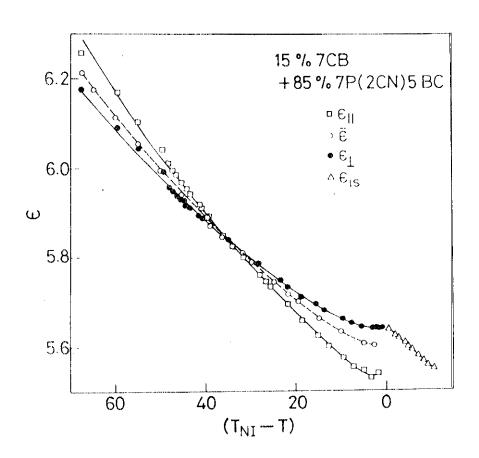


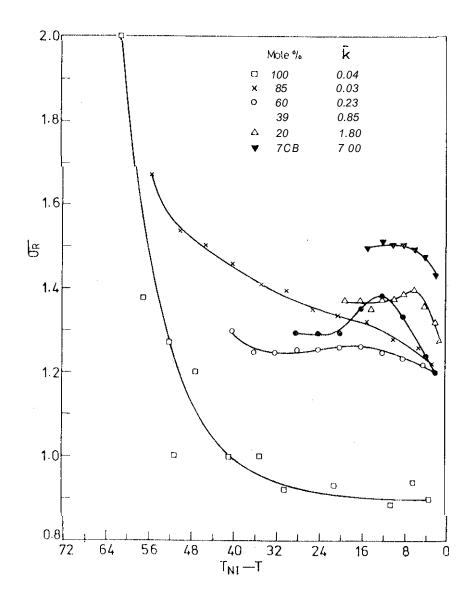
FIGURE 14

Temperature dependence of low frequency dielectric constants of a mixture of 85 mole per cent of 7P(2CN)5BC with 15 mole per cent of 7CB.

the skewed cybotactic structure in this mixture. conductivity anisotropy ( $\triangle o$ ) of these systems have been investigated for a range of frequencies from 300 Hz to In 7P(20N)5BC. Ac changes sign becoming negative 30 KHz. at higher temperatures for all frequencies investigated (fig. 15), again a consequence of the lowering of the tilt angle of the cybotactic groups. Further, at lower temperatures, the influence of the c., relaxation leads to an increase in the measured value of the conjuctivity as a function of frequency. 20 Unlike in normal nematics, however, both a,, and o, of 7P(2(M)5BC increase with frequency, practically by similar magnitudes, which is again a consequence of the skewed cybotactic structure in the medium. Xn mixtures with relatively large mole fractions of 708, the temperature variations of the conductivity anisotropies are governed by those of the strengths of cybotactic groups with relatively low tilt angles.

Some of the results discussed in this thesis are reported in the following publications.

Some unusual properties of 4-n-decylphonyl-3'methyl-4'-(4"-nitrobenzoyloxy)benzoate.
N.V. Madhusudana, B.S. Srikanta and M. Subramanya Raj Urs.
Molecular Crystals & Liquid Crystals Letters, <u>S2</u>. 25
(1982).



FIU.15: Temperature variations of the ratio of conductivities (at 3.592 Hz) in the nematic phase of the systems studied. The numbers appearing immediately after the symbols are the mole percentages of 7P(2CN)5BC and the next number is the mean specific conductivity  $\mathbb{R} \times 10^9$  (ohm cm)<sup>-1</sup> at 42°C.

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  N.V. Madhusudana. B.S. Srikanta and M. Subramanya
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