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## Liquid Crystals

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# A comparative study of the properties of the liquid crystalline compounds 5O.5 and 5O.6 in solid, solution and thin film forms by laser Raman spectroscopy

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Raman spectroscopic studies of the liquid crystalline compounds 5O.5 and 5O.6 revealed that the two aromatic rings in the core are in different environments with distorted electron cloud distributions, and the behaviour of each of these rings is independent of the other. Increased chain length results in the development of strain on the molecules. During the SmF–SmB phase transition there is a slight transfer of charge, along with an increase in the freedom of the individual molecules. Our experiments also reveal that the molecules are strained in the crystalline state and adopt some preferred orientations in free-standing films. There is also a tendency towards the elongation of the molecules along the C=N– linkage in solution for 5O.6. Raman spectra of 5O.6 in free-standing films have shown dramatic changes compared with the spectra recorded in the bulk.

## 1. Introduction

The orientational and translational ordering of the molecules in liquid crystals give rise to a rich variety of mesophases. One of the most interesting groups of liquid crystalline compounds is formed by the *N*-(4-*n*-alkyloxybenzylidene)-4'-*n*-alkylanilines whose molecules contain a Schiff's base central linkage and which are widely known as the *nO.m* series [1–3]. Of the *nO.ms*, the *N*-(4-*n*-pentyloxybenzylidene)-4'-*n*-alkylanilines (5O.ms) are a fascinating series because the compounds having intermediate values of *m*, namely 5O.5 and 5O.6, exhibit a rich variety of smectic mesomorphism in addition to the presence of a nematic phase. The lower homologues of the series, such as 5O.2, exhibit nematic and crystal G phases while the higher homologues show nematic, smectic A and smectic B phases. The phase sequence of the compounds 5O.5 and 5O.6 is N–SmA–SmC–SmF–G and N–SmA–SmC–SmB–SmF–G, respectively. This particular series of compounds also attracts much attention since the smectic A–nematic tricritical point among *nO.m* compounds is observed in this series [4]. Further interest in this series concerns the large variation of tilt angle (0° to 23° within a temperature range of 4°C) seen in the smectic C phase of 5O.5 [5] along with the existence of the smectic F phase in 5O.5 and 5O.6 (besides 9O.4),

which is an extremely rare phase in the *nO.m* family of compounds. Although the phase transitions and structural changes in the *nO.m* compounds have been studied extensively using various experimental techniques, a detailed spectroscopic analysis to probe the microscopic details of their different phases has not been reported, to the best of our knowledge.

Raman spectroscopy has proved to be a very powerful technique in the investigation of the vibrational dynamics of liquid crystalline compounds. The spectra–structure correlation, as well as the relationship between structural changes during phase transitions and the corresponding changes in the Raman spectra, is of particular interest. These relationships have been used [6–13] to obtain information on liquid crystals at the molecular level such as, for example, molecular orientation/rotation [14], and inter/intra-molecular interactions [15, 16].

Our earlier Raman spectroscopic studies on 5O.5 provided evidence for the existence of a lateral dipole moment at the core of the molecule [17]. We have seen already that 5O.5 and 5O.6 show the phase sequences N–SmA–SmC–SmF–G and N–SmA–SmC–SmB–SmF–G, respectively. Thus the increase by just one CH<sub>2</sub> group in the terminal alkyl chain of 5O.5 induces an orthogonal phase (smectic B) between two tilted phases (smectic C and smectic F). In an attempt to understand the origin of this behaviour at the molecular level, we have undertaken

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a comparative study of the properties of 5O.5 and 5O.6 using laser Raman spectroscopy. We also report here the results of Raman studies in the solution state of 5O.6. Some preliminary results on free standing films of the compound 5O.6 are also discussed.

## 2. Experimental

The compounds 5O.5 and 5O.6 were synthesized according to the literature procedure [4]. The samples obtained were repeatedly crystallized from absolute ethanol until phase transition temperatures were found to be constant. The transition temperatures were obtained using differential scanning calorimetry (Perkin-Elmer DSC7) and a polarizing microscope equipped with a Mettler FP82 hot stage and FP900 control unit, and were found to be in excellent agreement with values reported in the literature. The Raman spectra of the samples were recorded using a Spex Ramalog 1403 double monochromator equipped with an RCA-31034 photomultiplier tube and a CCD detector, in the  $1100\text{--}1250\text{ cm}^{-1}$  and  $1550\text{--}1650\text{ cm}^{-1}$  regions. The spectrometer control and data acquisition were achieved using DM3000 software

and a Spex Datamate, which is an 8-bit dedicated microcomputer. As the changes in the wave numbers of bands for different samples were small, we took the extra precaution of estimating the shifts by superimposing the different Raman spectra. The reported relative frequency shifts are accurate to within  $\pm 0.2\text{ cm}^{-1}$ . A Spectra-Physics Model 165-09  $\text{Ar}^+$  laser provided the excitation line at  $4880\text{ \AA}$ . To obtain the spectra for the bulk, the sample was confined in a quartz capillary tube of  $0.8\text{ mm}$  diameter sealed at both ends. Temperature resolution of the measurements was  $\pm 0.5^\circ\text{C}$ . A molar solution of the sample in  $\text{CS}_2$  was prepared to record the spectra in solution.

Thin film studies were carried out on free standing films of 5O.6. The free standing films were drawn across a conical hole made in a slab of hylam as well as in a glass slab. Free-standing films of the liquid crystal were drawn on the surface of the hylam using a spreader in the smectic A phase of the sample and were allowed to stabilize for some time. It is known that the molecular alignment in such films is homeotropic [18]. The construction of the thin film cell is shown in figure 1. To

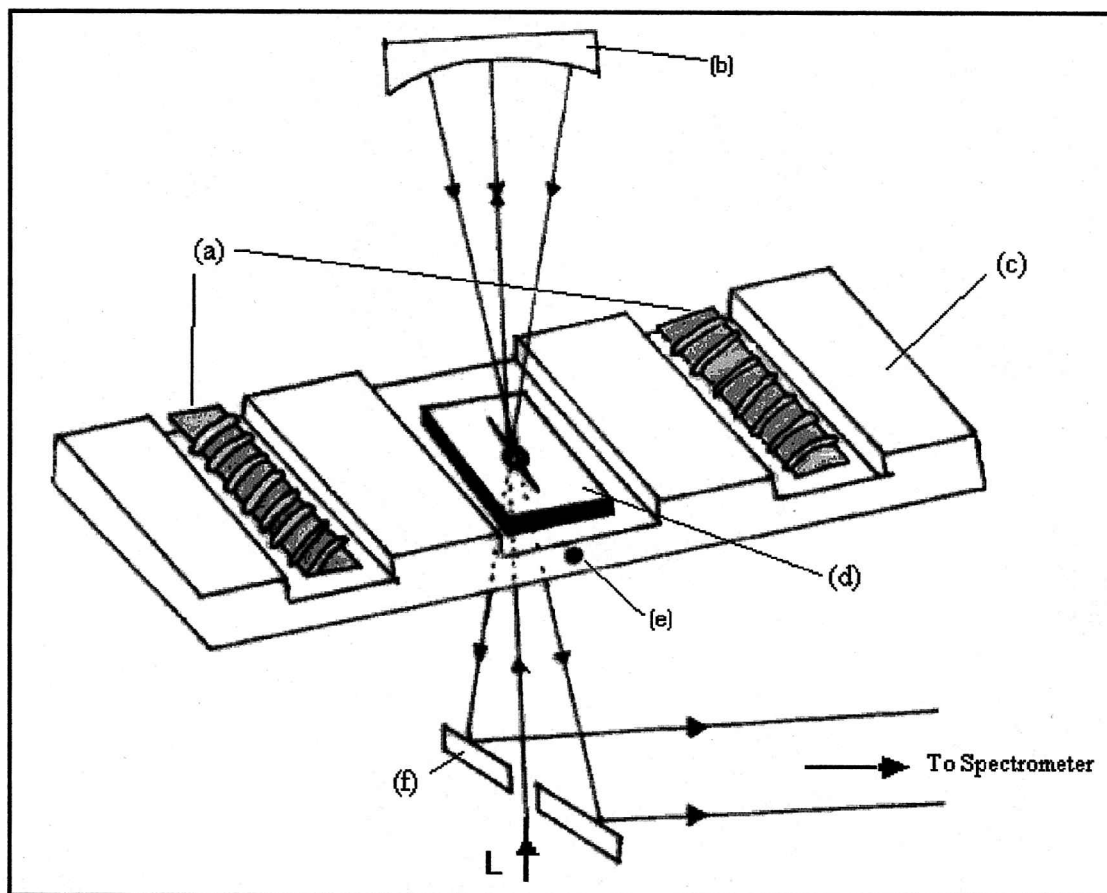
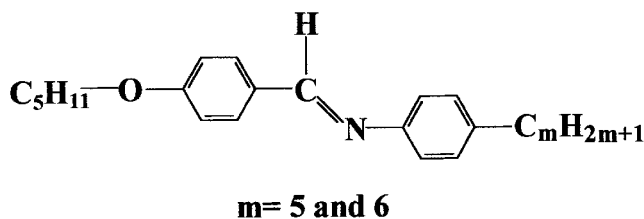


Figure 1. High temperature cell for the study of liquid crystals as free standing films. (a) Heating coils, (b) mirror, (c) aluminum base, (d) hylam surface with conical hole at the centre, (e) hole for the insertion of thermocouple, (f) mirror with a hole at the centre for  $180^\circ$  scattering geometry. Arrows (L) indicate the laser beam.

the best of our knowledge, no reports of free-standing films drawn using hylam slab (a laminate composite used in electrical printed circuit boards) are available in the literature. We found that with a hylam surface, not only drawing the films was easier, but also that the stability of these films was found to be excellent. The thickness of the films was estimated using an interference pattern from the transmitted portion of the laser beam.

### 3. Results and discussion

The molecular structure of the compounds 5O.5 and 5O.6, phase sequences and transition temperatures are given below.



5O.5:  
Cr 28 G 47 SmF 49 SmC 53.1 SmA 54.4 N 77.8 I (°C)

5O.6:  
Cr 36 G 38.4 SmF 42.4 SmB 50 SmC 51.8 SmA 60.3 N 72.8 I (°C)

Clearly, both molecules are composed of two regions. The two phenyl rings along with  $-C(H)=N-$  linkage forms the mesogenic core while the two terminal alkyl chains constitute alkyl segments. Raman spectra were obtained in the  $1100$ – $1250\text{ cm}^{-1}$  and  $1550$ – $1650\text{ cm}^{-1}$  regions as both these regions contain bands related to the core whose behaviour is the subject of our interest.

#### 3.1. Comparison of Raman Spectra of 5O.5 and 5O.6 in the crystalline state

It was already observed in 5O.5 [16] that the oxygen present in this molecule severely distorts the electron clouds in various parts of the molecule and this distortion is dependent on the molecular conformations as well as the type of phase. The analysis of the spectra recorded in the  $1140$ – $1220$  and  $1550$ – $1650\text{ cm}^{-1}$  regions for 5O.5 have already been reported [17]. Raman spectra in the bulk sample of 5O.6 at different temperatures were recorded in the  $1100$ – $1250$  and  $1550$ – $1650\text{ cm}^{-1}$  regions for comparative studies over the entire mesophase region.

In the crystalline state, both 5O.5 and 5O.6 crystallize in a  $c$ -centered monoclinic lattice [19, 22–24] with a two-fold axis along the  $b$ -axis and molecular orientation along the  $c$ -axis. The  $c$ -axis is approximately half the molecular length. Therefore the molecular length extends along the  $c$ -axis and a layer forms along the  $b$ -axis. Given that the  $b$ -axis is two-fold and the molecules are staggered, their most probable conformation is that in

which the phenyl rings are oriented along the  $ac$  plane. Thus, we can easily visualize that the alkyl chains of the adjacent rings will be closer along the  $c$ -axis and the phenyl rings will be closer along the  $b$ -axis.

As in 5O.5 that we have investigated earlier, the Raman spectrum of the bulk sample of 5O.6 also showed a triplet in the  $1155$ – $1170\text{ cm}^{-1}$  region. Similarly the doublets in the  $1570$ – $1580$  and  $1594$ – $1600\text{ cm}^{-1}$  regions observed for 5O.5, were also seen for 5O.6. But for 5O.6, the doublets and triplets are more clearly visible and the splittings become sharper, see figure 2, spectra (a) and (b).

The origin of these triplets and doublets has been discussed in detail in our earlier paper [17]. A tentative assignment of all the peak positions made on the basis of the expected group frequencies and reported assignments for similar Schiff's base compounds, in the bulk sample, solution state and free standing films for 5O.6 are given in table 1 [15, 20, 21]. The spectra data for 5O.5 is also included in table 1 for comparison. The only difference between 5O.5 and 5O.6 is an extra  $\text{CH}_2$  group in one of the alkyl chains of 5O.6. It should be noted that both the alkyl chains in the earlier sample were equal in length whereas in the present sample the alkyl chain adjacent to the oxygen atom is shorter than the other. This subtle change in the case of 5O.6 results in the effect of the oxygen atom becoming more dominant, as the  $\pi$ -electron cloud is now more severely distorted; this affects the charge distribution of the phenyl rings adjacent to it. These changes are accompanied by small but definite relative shifts in the peak positions of all the representative bands towards lower wave numbers by a few wave numbers (table 1) indicating a decrease in the energy of the respective bond undergoing vibration (a loosening of the bond). This is expected to be due to the increased orientational freedom of the long alkyl chain due to the decrease in intermolecular interactions which may put strain (by increased stretching along the molecular axis) on the whole core region.

Apart from frequency shifts, changes in the band width of certain Raman bands also indicate the magnitude of disorder in the system. We have analysed our results by taking into account the shift in band positions as well as changes in the line width of Raman bands. Given the accuracy of the measurements of peak positions in these experiments and in our previous work, the expected shift in peak positions due to an increase or decrease in the number of methylene units in the terminal alkyl chains is only of the order of a few wave numbers [15]. Therefore, the changes observed in the modes associated with the core region suggest that the core is also affected by the increased chain length. One can rationalize these observations in the following way: as the chain length increases, the  $\text{CH}_2$  groups will come closer along the  $c$ -axis and will be stabilized in a minimum energy

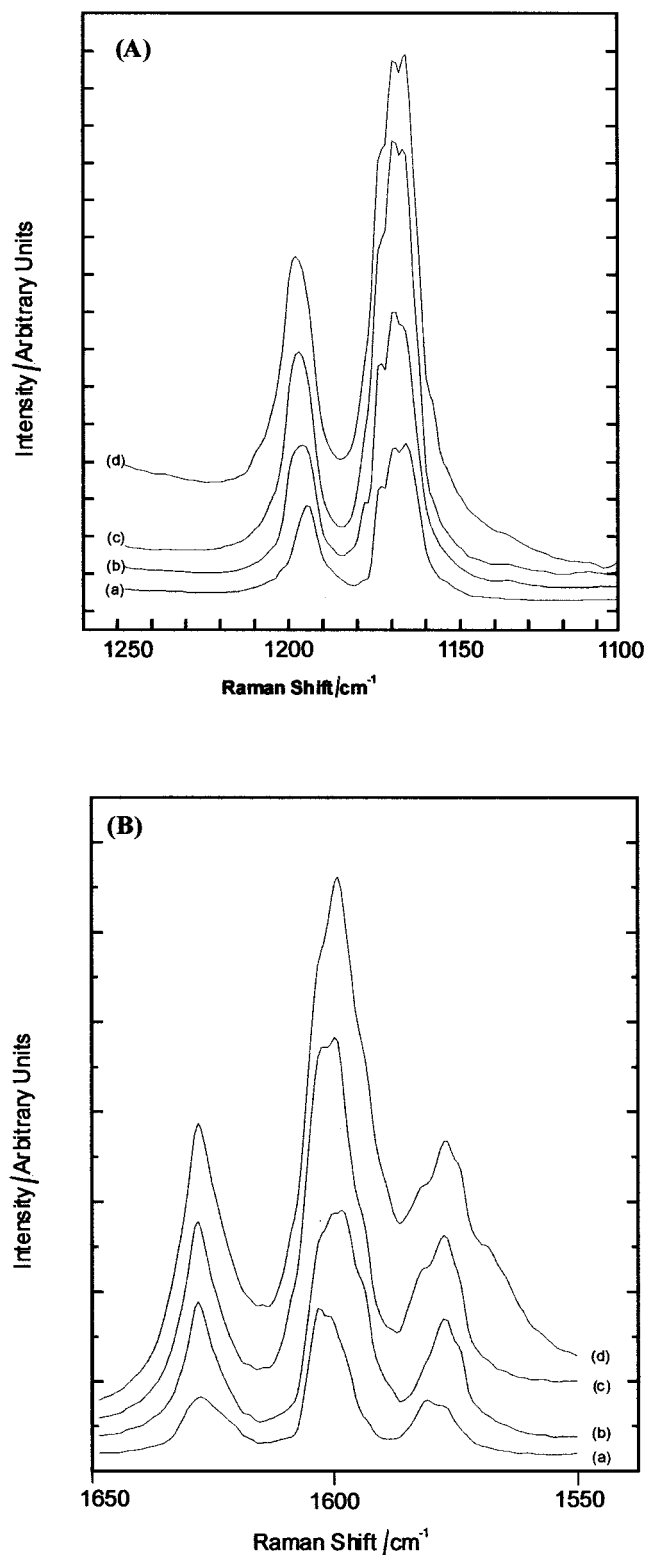


Figure 2. Raman spectra in the 1100–1250 cm<sup>-1</sup> (A) and 1550–1650 cm<sup>-1</sup> (B) regions in different phases. (a) 5O.5 crystalline phase in bulk, (b) 5O.6 crystalline phase in bulk, (c) 5O.6 in CS<sub>2</sub> solution, (d) 5O.6 in free-standing thin films.

conformation due to steric hindrance, whatever the conformation may be, i.e. *trans* or *gauche*. Moreover, as a result of the increased stress imposed by the long alkyl chains upon the backbone, the  $\sigma$ -electron cloud over the phenyl ring bond may also distort significantly. With such constraints the two benzene rings are unable to orient themselves in a single plane, but there is a possibility that rotation occurs along the C–N single bond and the molecule is stabilized in a minimum energy conformation.

### 3.2. The solution state

In order to probe whether there is any appreciable change in intermolecular interactions between the solid and liquid crystalline states on increasing the chain length by one methylene unit, we have measured the spectra of 5O.6 in solution in carbon disulphide. The solution state spectrum is marked by some very interesting changes. Previous studies of similar Schiff's base compounds [15] without an oxygen atom (such as, for example, TBBA and TBDA) have shown that the peak positions of all the core-related bands shift to lower wave numbers compared with their counterparts in the solid state (figure 2). The reverse is observed however, for 5O.6. The bands arising due to the C–O and the aromatic C–N stretching modes show an upward shift of peak positions compared with the crystalline state. The bands arising due to the phenyl ring modes, however, are almost unchanged. The band arising due to the C=N stretching mode shifts to a lower wave number. This shows that the major change that occurs in the solution state is that the strain on the alkyl chains and on the linking bonds of the phenyl rings are relaxed and that there is a tendency towards the elongation of the individual molecules. As a result of this, there is increased strain on the C=N bond resulting in the redistribution of the charges around this region. Also accompanying this, are changes in peak position of the C=N mode (4 cm<sup>-1</sup>) and in the peak position of one of the aromatic in-plane bending modes towards higher wave numbers. This occurs since the change in peak position is observed in the mode related to the phenyl ring adjacent to the oxygen atom, which gains charge density.

In the solution state, it may be noted that the restrictions on movement of molecules are lifted because now the molecule need not be confined to a lattice. Also, intermolecular interactions being almost negligible in solution, each segment of the molecule now has more rotational and orientational freedom. The red shift of the representative bands from solid to solution state, along with the increase in line width of these bands, indicates that the strain imposed in the crystalline state is eased making the respective bonds stronger and at the same time increasing the orientational disorder.

Table 1. Peak positions ( $\text{cm}^{-1}$ ) and linewidths ( $\text{cm}^{-1}$ ) of the various core-related modes of 5O.5 and 5O.6 along with assignments in the crystalline state, solution and in bulk. PP = peak position, LW = linewidth.

| Assignments                                   | 5O.5 (bulk) |     | 5O.6 (bulk) |      | 5O.6 (solution) |      | 5O.6 (thin films) |      |
|---|-------------|-----|-------------|------|-----------------|------|-------------------|------|
|   | PP          | LW  | PP          | LW   | PP              | LW   | PP                | LW   |
| Aromatic C–H in plane bending mode            | 1162        | 8.9 | 1160        | 8    | 1161            | 9.4  | 1158              | 9    |
| Aromatic C–H in plane bending mode            | 1167        | 5.5 | 1165        | 5.0  | 1165            | 6.5  | 1164              | 5.7  |
| C–O stretch                                   | 1171        | 5.9 | 1169        | 6.8  | 1172            | 6.9  | 1178              | 6.0  |
| Aromatic C–N stretching mode                  | 1194        | 7.4 | 1192        | 8.4  | 1195            | 8.2  | 1201              | 8.0  |
| Quadrant stretching mode of the aromatic ring | 1572        | 7.8 | 1571        | 6.53 | 1573            | 10.3 | 1569              | 9.8  |
| Quadrant stretching mode of the aromatic ring | 1575        | 7.3 | 1574        | 6.8  | 1576            | 10.0 | 1572              | 9.1  |
| Quadrant stretching mode of the aromatic ring | 1594        | 6.2 | 1593        | 9.0  | 1595            | 9.5  | 1592              | 8.7  |
| Quadrant stretching mode of the aromatic ring | 1598        | 7.7 | 1597        | 8.6  | 1599            | 8.7  | 1596              | 10.1 |
| C=N stretching mode                           | 1627        | 7.3 | 1626        | 8.4  | 1622            | 8.6  | 1628              | 9.3  |

### 3.3. The smectic F–smectic B phase transition

The changes in the Raman spectral parameters (peak position, line width and integrated intensity of various core-related modes) for 5O.6 are found to be almost identical to those seen for 5O.5 at the G–SmF, SmA–N and N–I phase transitions. As the temperature range of the SmC phase is very small ( $1.8^\circ\text{C}$ ), the spectral changes at the SmC–SmB transition could not be monitored. Therefore, we discuss here the spectral changes at the SmF–SmB transition only.

The SmF–SmB phase transition occurs at  $42.4^\circ\text{C}$ . The SmF phase has a *c*-centered monoclinic lattice with strong in-plane short range positional correlations and almost negligible inter-layer positional correlations. The molecules in this phase are tilted with respect to the layer planes and are packed in pseudo-hexagonal clusters. In the SmB phase the molecules form stacks of interacting hexatic layers with strong in-plane short range positional correlation. The interlayer positional correlation is almost non-existent in this phase. In this phase, the molecules are not tilted but are orthogonal with respect to the layer planes. Thus the only difference between the SmF and SmB phases is that the molecules are tilted in the former whereas they are orthogonal in the latter. When the transition from the SmF to the SmB phase takes place, the tilted molecules in the SmF phase become orthogonal. Peak positions, line widths and the integrated intensities of various core-related Raman modes of 5O.6 in the SmF and SmB phase are compared in table 2. This suggests that there is no major change in either the peak positions or in the line widths of any of the bands. Very subtle increases in the peak positions and line widths of almost all the bands indicate a slight increase

Table 2. Comparison of the various Raman parameters of the core-related modes of the 5O.6 molecules in the SmF and the SmB states. PP = peak position, LW = linewidth, II = integrated intensity.

| Sr. No | Smectic F state         |                         |       | Smectic B state         |                         |       |
|--------|-------------------------|-------------------------|-------|-------------------------|-------------------------|-------|
|        | PP/<br>$\text{cm}^{-1}$ | LW/<br>$\text{cm}^{-1}$ | II    | PP/<br>$\text{cm}^{-1}$ | LW/<br>$\text{cm}^{-1}$ | II    |
| 1      | 1161                    | 6.96                    | 29.77 | 1161                    | 6.08                    | 28.82 |
| 2      | 1166                    | 6.73                    | 18.07 | 1166                    | 6.23                    | 23.80 |
| 3      | 1169                    | 5.73                    | 24.67 | 1170                    | 6.20                    | 24.31 |
| 4      | 1193                    | 9.43                    | 27.48 | 1194                    | 9.15                    | 23.06 |
| 5      | 1570                    | 7.18                    | 19.98 | 1571                    | 7.60                    | 19.34 |
| 6      | 1574                    | 5.13                    | 42.06 | 1574                    | 4.70                    | 38.85 |
| 7      | 1592                    | 7.45                    | 17.27 | 1593                    | 7.50                    | 20.96 |
| 8      | 1596                    | 9.66                    | 7.41  | 1597                    | 9.10                    | 5.98  |
| 9      | 1625                    | 10.40                   | 13.26 | 1626                    | 10.60                   | 14.87 |

in the freedom of the individual molecules in the SmB phase. The integrated intensity, on the other hand, shows considerable increases for those bands which are related to the phenyl ring adjacent to the oxygen atom. Similarly, a decrease is observed in the integrated intensity for the bands related to the C–N stretch. This indicates that due to the change in the alignment of the molecules from being tilted in the SmF phase to orthogonal in the SmB phase, some conformational changes of the phenyl ring around the single bond occur, resulting in charge transfer from the nitrogen atom towards the phenyl ring adjacent to the oxygen atom. This charge transfer is possible due to the lower electronegativity of nitrogen (2.5) compared with that of oxygen (3.5) permitting the mesomeric delocalization of the  $\pi$ -electron cloud.

### 3.4. Thin films

Free-standing homeotropically aligned thin films of 5O.6 in the SmA phase were obtained on a hylam surface using a custom-built high temperature cell. In free-standing films the molecules align parallel to the direction of the incident radiation. In the absence of an applied electric field, the molecular orientation varies continuously over the film surface in 'swirl patterns' [25]. These patterns are interrupted by a small number of point defects which was observed by focusing the transmitted portion of the laser beam onto a screen. These patterns stabilized after 15–20 min. In the SmA phase the molecules are arranged in equidistant layers and form a one-dimensional periodic structure in a direction perpendicular to the layers. There is no translational order within the layers, which is similar to the case of two-dimensional fluids. Orientational order is certainly found to exist, and the molecular long axes are on average oriented perpendicular to the layers. In the films that we have drawn in the SmA phase, the probability that the average long molecular axes are perpendicular to the respective layers is further enhanced. As stated earlier, these molecules are found to possess a lateral dipole moment. In the absence of an electric field, these dipoles can be assumed to be oriented randomly in two configurations which for convenience, we term the up and the down states. Due to this random orientation no spontaneous polarization is observed.

When the Raman spectra of these molecules in the thin films were recorded, dramatic variations were noticed. The peak position of the bands related to the phenyl rings, both the in-plane C–H stretching modes as well as the quadrant stretching modes, showed a shift in the peak positions towards lower wave numbers [figure 2 spectra (*d*)]. The C–O stretching mode at  $1169\text{ cm}^{-1}$  and the C–N stretching mode at  $1192\text{ cm}^{-1}$  showed large shifts in peak positions towards higher wave numbers. Surprisingly, however, the C=N stretching mode at  $1626\text{ cm}^{-1}$  showed only a slight shift in peak position to higher wave numbers. These changes indicate that the central linkage of the core remains almost unaffected during the formation of the free-standing thin films. There is a transfer of charge from the phenyl rings towards the alkyl chains. Such changes result from conformational changes in the alkyl chains and also twisting of the whole core around the single bonds at the ends of the core—facilitated by the attainment of the ordered layered structure in the homeotropic free standing thin films [26]. Further analysis of these spectra in comparison with the spectra of the bulk sample, as well as a study of the variation of the different Raman parameters with temperature in free-standing thin films of 5O.6 and 5O.5, are in progress.

### 4. Conclusions

Comparative studies of 5O.5 and 5O.6 suggest that the strain on the various molecular segments increases with an increase in the terminal chain length of the molecule, as is evident from the decrease of the peak positions for the higher homologue, 5O.6. In the solution state, although the strain on the molecule due to the lattice is reduced, there is a tendency towards elongation of the molecule resulting in an increase in the strength of all bonds except for a weakening of the C=N bond. In a thin film, the molecules are aligned perpendicular to the layers in a random bipolar orientation without significantly affecting the core.

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