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## Liquid Crystals

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# Influence of spacer and terminal group lengths on the smectic ordering of cholesterol-containing dimer liquid crystals

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The smectic properties of four series of dimer liquid crystals containing cholesteryl and biphenyl groups were investigated by polarization microscopy, DSC and X-ray diffraction. Compounds that contain the strongly dipolar cyanobiphenyl group exhibit a smectic layer spacing that is about 1.7 times the length of the molecule. Three series of alkoxybiphenyl-containing dimers exhibit two other smectic modifications. For short spacers, a smectic layer spacing is observed that is about the same length as the molecule and for long spacers the smectic layer spacing is about half the length of the molecule. In the latter cases the entropy change at the SmA–N\* transition is clearly larger than for the compounds with the other smectic modifications. One or two compounds in each alkoxybiphenyl-containing series, that have similar spacer length and terminal group length and have an odd number of flexible units in the spacer, exhibit only a chiral nematic phase, and no smectic phase.

## 1. Introduction

Dimer liquid crystals (LCs) form a class of LCs in which two mesogenic units are connected by a flexible spacer. These compounds show several intriguing properties [1–3]. They exhibit a strong odd–even effect in the phase transitional properties depending on the parity of the spacer. The members of a dimer series with an even spacer, for example, have higher isotropization temperatures, higher associated enthalpy effects and better ordered phases than the members with an odd spacer. For chiral cholesterol-containing dimers it was found that the selective reflection wavelengths of the chiral nematic phase are higher for the members with an even spacer than for members with an odd spacer [4–6]. Another interesting phenomenon of dimer LCs with smectic phases is the variation found in the smectic layer spacings. Within the same series the smectic layer spacing can vary depending on the relative lengths of both spacer and terminal groups.

For non-symmetrical dimer LCs with two aromatic mesogenic units it has been found that two different SmA modifications can exist depending on the lengths of the spacer and the terminal alkyl chain [1, 2, 7, 8]. For a certain length of terminal alkyl group and shorter

spacers, an interdigitated SmA (SmA<sub>d</sub>) phase was found, with a smectic layer spacing  $d$  of  $> 1.5$  times the length  $l$  of the molecule. In these layers the more dipolar mesogenic units have an antiparallel arrangement. When the spacer is longer than the terminal alkyl groups an intercalated SmA (SmA<sub>c</sub>) phase is observed with a  $d/l$  ratio of about 0.5. In this phase, layers are present with equal amounts of both mesogenic units and the spacers form the connection between the mesogenic units in the different layers. When spacer and terminal alkyl chain lengths are about equal, neither smectic modification is favourable; the smectic behaviour disappears and only a nematic phase is found. For symmetrical dimers with the mesogenic units connected to the spacer through ether groups, smectic phase behaviour was found for terminal alkyl groups longer than half the spacer length. In these cases the smectic layer spacings were found to be about equal to the molecular length [9]. Recently, this behaviour was also found for some non-symmetric dimers, which differed only in the lengths of their terminal alkyl groups [10].

For dimers with cholesteryl moieties, however, slightly different behaviour has been found [11–16]. Depending on spacer, type of aromatic mesogen and terminal alkyl chain length, three different smectic modifications were again observed. The smectic layer spacing observed using X-ray diffraction (XRD) indicates intercalated

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smectic phases with a  $d/l$  ratio of about 0.5 when the spacer is longer, and smectic phases with a  $d/l$  ratio of about 1, when the spacer is shorter than the alkyl terminal chain. When spacer and terminal alkyl groups are about equal in length, so-called incommensurate or frustrated smectic phases are sometimes found for dimers with Schiff's base-containing aromatic mesogenic units. Recently, interdigitated phases with a  $d/l$  ratio of about 1.5 were found for cholesteryl dimers with electron-withdrawing groups at the end of the aromatic mesogenic unit [16].

For a series of biphenyl and cholesterol-containing dimer liquid crystals that we recently investigated, we observed smectic phases for terminal group lengths both shorter and longer than the spacer length, but no smectic behaviour for a compound with a terminal group length equal to the spacer length [17]. This result prompted us to study the phase behaviour of these compounds and of some related series of compounds in more detail.

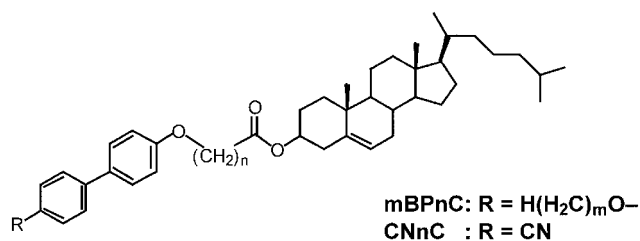
## 2. Experimental

The synthesis and characterization of the compounds was carried out by methods described earlier [4, 5, 17]. Melting points and thermal phase transition temperatures were determined, and optical inspection of the liquid crystalline phases made, on samples between glass slides using an Olympus BH-2 polarization microscope equipped with a Mettler FP82HT hot stage controlled by a Mettler FP80HT central processor. Differential scanning calorimetry (DSC) thermograms were obtained on a Perkin Elmer DSC-7 system. The entropy changes at the phase transition temperatures are expressed as  $\Delta S/R$ , in which  $\Delta S$  is calculated from  $\Delta S = \Delta H/T$ .  $\Delta H$  is expressed in  $\text{J mol}^{-1}$  and  $T$  is the corresponding phase transition temperature in Kelvin. XRD measurements were made on the samples employing a Bruker HSTAR area detector using monochromatic  $\text{CuK}_\alpha$  radiation. The temperature was controlled by an oven which was built in-house.

## 3. Results and discussion

### 3.1. General properties

Three series of cholesteryl  $\omega$ -(4'-alkoxybiphenyl-4-yloxy)alkanoates were synthesized (see the scheme).



Scheme.

All the compounds gave single spots on thin layer chromatography, correct elemental analyses and NMR spectra which were in agreement with the structure. The liquid crystalline phases were identified by polarizing optical microscopy (POM) and XRD. The chiral nematic phases were easily identified by the typical texture of this phase and the observation of selective reflection of visible light from an aligned sample between glass slides. The SmA phases gave focal-conic textures or, when sheared between glass slides, a dark homeotropic texture was observed, indicative of a non-tilted smectic phase. XRD gave one or two rings in the small angle region, and a diffuse ring in the large angle region, typical for the SmA phase. The thermotropic properties of the compounds as obtained by DSC are given in the table.

As seen in figures 1 and 2, the three series of compounds *mBPnC* exhibit a strong alternation with  $n$  of the isotropization temperatures and of the corresponding entropy changes ( $\Delta S/R$ ) at the N\*-I transition. This well known odd-even effect is related to the fact that dimer LCs with an even number of flexible units in the spacer between the rod-like mesogenic groups give better ordered nematic phases than those with an odd number of flexible groups in the spacer [1-3]. Therefore, their isotropization temperatures and corresponding heat

Table. Phase transition temperatures and smectic layer spacings  $d$  of the liquid crystal dimers series *mBPnC*. The reduced transition entropies ( $\Delta S/R$ ) are given in parenthesis.

Compound	M.p./°C	SmA-N*		N*-I		$d/\text{\AA}$
		$T/^\circ\text{C}$	$\Delta S/R$	$T/^\circ\text{C}$	$\Delta S/R$	
4BP3C	171	169	(0.18)	215	(1.02)	40
4BP4C	104	—	—	152	(0.33)	—
4BP5C	118	171	(0.49)	194	(1.24)	20
4BP6C	92 <sup>a</sup>	—	—	142	(0.32)	—
4BP7C	112 <sup>a</sup>	166	(0.63)	177	(1.33)	21
4BP10C	99	130	(0.38)	144	(0.62)	22
5BP3C	174 <sup>a</sup>	188	(0.13)	206	(1.07)	40
5BP4C	117	126	(0.09)	148	(0.39)	40
5BP5C	143 <sup>a</sup>	169	(0.56)	187	(1.35)	20
5BP6C	86 <sup>a</sup>	—	—	141	(0.34)	—
5BP7C	95	165	(0.77)	171	(1.43)	21
5BP10C	99	131	(0.36)	142	(0.61)	23
6BP3C	160	198	(0.28)	207	(1.15)	<sup>b</sup>
6BP4C	127	133	(0.17)	148	(0.40)	43
6BP5C	136	162	(0.12)	181	(1.20)	44
6BP6C	103 <sup>a</sup>	—	—	123	(0.34)	—
6BP7C	110	161	(0.65)	168	(1.52)	22
6BP10C	95 <sup>a</sup>	130	(0.64)	138	(0.67)	23

<sup>a</sup> These compounds may exist in other crystalline forms with lower melting points: 4BP6C 72°C; 4BP7C 89°C; 5BP3C 159°C; 5BP5C 130°C; 5BP6C 73°C; 6BP6C 96°C; 6BP10C 72°C.

<sup>b</sup> Not measured.

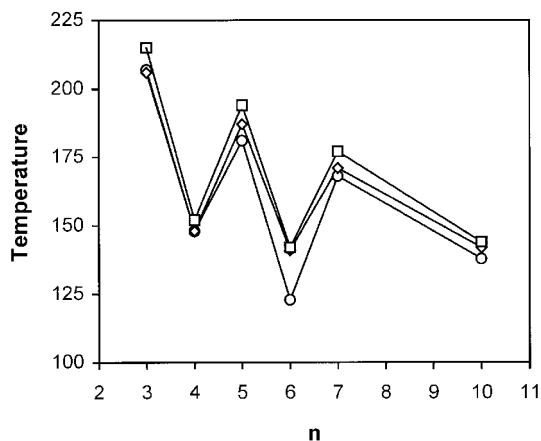


Figure 1. N\*-I transition temperatures of the series 4BPnC (□), 5BPnC (◇) and 6BPnC (○) as a function of spacer length  $n$ .

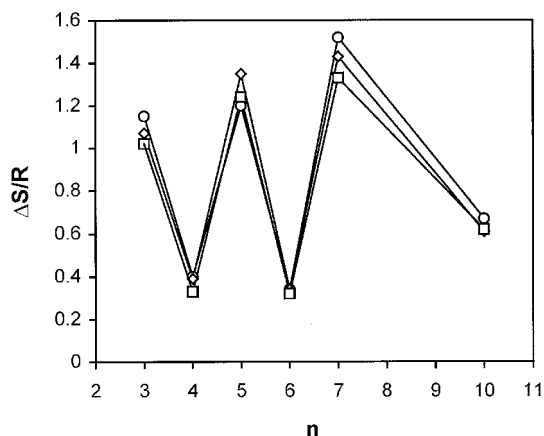


Figure 2. Entropy change  $\Delta S/R$  at the N\*-I transition of the series 4BPnC (□), 5BPnC (◇) and 6BPnC (○) as a function of spacer length  $n$ .

effects are higher than for the members in the series with an odd spacer. These findings are comparable to the results obtained for the cyanobiphenyl-containing series CNnC investigated earlier [4, 5].

### 3.2. Smectic properties

First, the smectic properties of the three series  $mBPnC$  ( $m = 6, 5$  and  $4$ ) and the related series CNnC will be discussed. As described before, no smectic phase is observed for  $n = 6$  in the series 6BPnC [17]. This compound forms a chiral nematic phase that crystallizes upon cooling to about  $70^\circ\text{C}$  without forming a smectic phase. For the other compounds the SmA-N\* transition temperatures follow the same alternating trend as the N\*-I transition temperatures. For an even number of flexible units ( $n + 3$ ) in the spacer ( $n = 3, 5$  and  $7$ ) they are higher than for the compounds with an odd number ( $n + 3$ ) of flexible units in the spacer ( $n = 4$  and  $10$ ).

As judged by POM, the transition from SmA to chiral nematic is different for the compounds with a short spacer than for the compounds with a longer spacer. For short spacers, the transition is preceded by the growth of curved lines in a homeotropic sample, whereas for longer spacers such lines are absent. This indicates that the transition is accompanied by an intermediate twist grain boundary (TGB) phase [15–20]. For the compounds with the longer spacers, 6BP7C and 6BP10C, this transition is more abrupt and no indication of an intermediate TGB phase was found. On DSC, the transitions that are accompanied by a TGB phase are broadened, but no separate peaks were observed. Interestingly, the total entropy change at the SmA-N\* transition (including that from the TGB phase) for the compounds with the short spacers is much smaller than for the compounds with the long spacers (figure 3 and the table). POM and DSC give no indication for the formation of incommensurate phases, i.e. no small transition peaks over a broad temperature range are observed. As described before, blue phases were observed upon slow cooling from the isotropic phase for some compounds with a small  $\Delta S/R$  value of the N\*-I transition [17].

By XRD, layer spacings of about  $43 \text{ \AA}$  were obtained for  $n = 4$  and  $5$  and  $22 \text{ \AA}$  for  $n = 7$  and  $10$ , see figures 4(a) ( $n = 5$ ) and 4(b) ( $n = 7$ ). For short  $n$  this gives a  $d/l$  ratio of about 1 and for longer  $n$  the  $d/l$  ratio is about 0.5. The lengths  $l$  of the molecules are estimated from molecular models of the compounds in their most extended conformations. These results correspond quite well with results obtained for related compounds described by Hardouin *et al.* [11, 14] in which spacer length and end group length are varied.

For the series 5BPnC, a similar relation between mesogenic properties and spacer length is found as for the series 6BPnC. Like 6BP6C, compound 5BP6C shows no smectic phase (see the table). The main difference

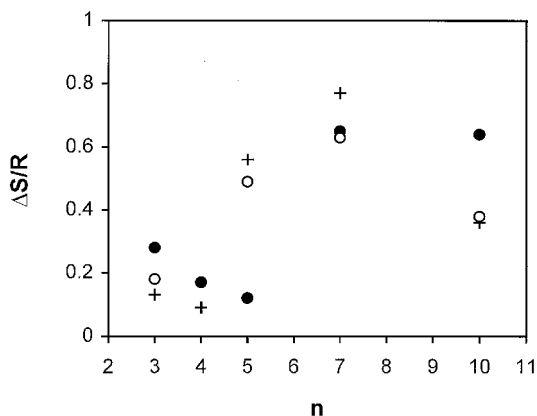


Figure 3. Entropy change  $\Delta S/R$  at the SmA-N\* transitions of the series 4BPnC (○), 5BPnC (+) and 6BPnC (●) as a function of spacer length  $n$ .

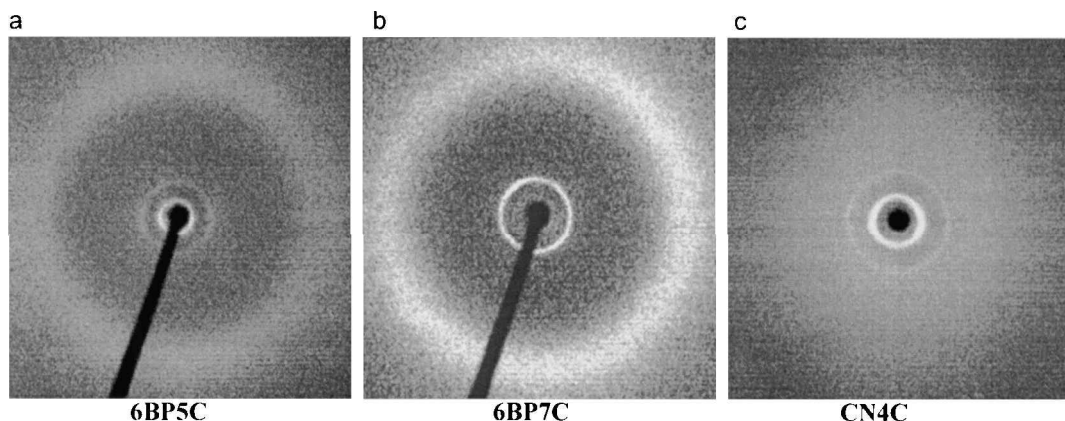


Figure 4. X-ray diffraction patterns of (a) 6BP5C, (b) 6BP7C and (c) CN4C in the SmA phase. For CN4C a smaller beam stop was used and the detector was placed at a larger distance from the sample.

between series 5BP $n$ C and 6BP $n$ C is that the  $\Delta S/R$  value of 5BP5C is now significantly larger than for 6BP5C and corresponds better with the values found for the compounds with longer spacers ( $n = 7$  and 10). From the layer spacing of 20 Å obtained for this compound by XRD, a  $d/l$  ratio of about 0.5 can be calculated, similar to the values for the compounds with longer spacers. Therefore, both the  $\Delta S/R$  value and the  $d$ -spacing of 5BP5C correspond with the values for compounds with the long spacers ( $n = 7$  and 10). For the compounds 5BP $n$ C with  $n = 3$  and 4, the  $d/l$  ratio is about 1.

Surprisingly, two compounds in the 4BP $n$ C series, namely those with  $n = 4$  and 6, show only chiral nematic and no smectic behaviour, whereas the compound with  $n = 5$  has a smectic phase. The layer spacing for the smectic phase of this compound is about 20 Å and the entropy change  $\Delta S/R$  at the SmA–N\* transition is rather large (0.49). In this series, only compound 4BP3C exhibits a small entropy change and a large  $d$ -spacing.

When the results from DSC measurements and POM for these three series of compounds are compared with those obtained for the dimer series CN $n$ C, large differences are apparent. For this latter series of compounds, all values of  $\Delta S/R$  at the smA–N\* transition are rather small (0.1–0.3) and they show no distinct trend with the parity of the spacer [4, 5]. Furthermore, all compounds in this series have SmA phases and the nematic window gradually increases with spacer length (see figure 5).

By XRD rather large smectic spacings of 58 Å for CN4C, figure 4(c), and 66 Å for CN5C are found for these compounds. These values correspond to interdigitated smectic phases with a  $d/l$  ratio of about 1.7 (the length of these molecules is estimated to be about 36 and 38 Å, respectively). Interestingly, this agrees well with the smectic ordering found recently for cholesteryl dimers with a strong electron-withdrawing substituent at

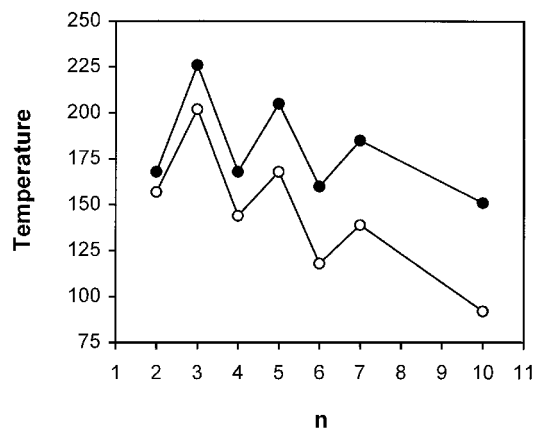


Figure 5. SmA–N\* transition temperatures (○) and N\*–I transition temperatures (●) of the series CN $n$ C as a function of spacer length  $n$ .

the terminus of the aromatic mesogen [16]. Reinvestigation of the SmA–N\* transition by POM showed that in this series all these transitions are accompanied by a TGB phase. Furthermore, upon slow cooling from the isotropic phase of the compounds CN2C, CN4C and CN6C, the typical platelet texture of a blue phase is found. This behaviour might be associated with the low heat effects for the N\*–I transitions, but also with the small helical pitch exhibited by these dimers with odd spacers [4–6, 17], as suggested by Luckhurst *et al.* [6].

### 3.3. Models for smectic ordering

Based on the observed  $d/l$  ratio, a packing model for the CN $n$ C dimers in the SmA phase can be derived (figure 6). In this model there is antiparallel overlap between the aromatic groups, as is often suggested for cyanobiphenyl LCs. The cholesteryl groups (which have a larger cross-sectional area than alkyl and aromatic

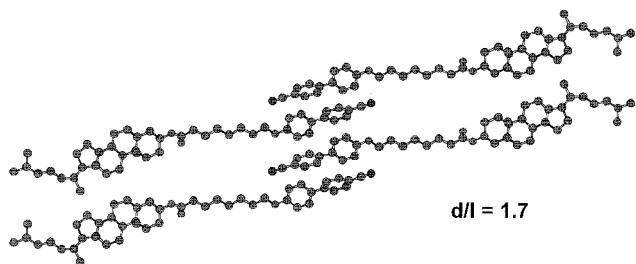


Figure 6. Schematic representation of the possible packing of CN7C molecules in the SmA phase (CN7C in the figure).

groups), the aromatic groups and the alkyl spacers form separate sublayers. It is easily seen that in this model longer spacers destabilize the smectic ordering due to their small cross-sectional area; this could explain the increase in the nematic window of this series with increasing spacer length.

For the three series of alkoxybiphenyl-containing dimers were found four compounds which lack a smectic phase. All of them have an odd number ( $n + 3$ ) of flexible units in the spacer between the two mesogenic groups. This indicates that when the spacer length  $n$  and terminal alkyl length  $m$  are about equal, and the compounds have an odd number of flexible units in their spacer, a smectic ordering is hampered. In this respect the behaviour is similar to that found for the non-symmetric dimers investigated by Luckhurst *et al.* [6, 7]. However, for their compounds the  $d/l$  ratio changes from about 1.8 to 0.5, whereas for ours the  $d/l$  ratio changes from 1.0 to 0.5 upon going from short to long spacers. It is realized that in our series of compounds only the alkyl terminal group of the aromatic mesogenic unit changes and the alkyl group of the cholesteryl mesogenic unit remains the same. It has, however, about the same length as the end groups of the aromatic mesogenic units used in our series.

From the three series it can also be deduced that a small entropy change at the SmA–N\* transition corresponds nicely with a large  $d$ -spacing, and a larger entropy change with a smaller  $d$ -spacing (see the table). This indicates that the SmA phase with a small  $d$ -spacing is more stable and that it is not favourable for the compounds with a short spacer. Hardouin *et al.* have performed detailed XRD and molecular mechanics calculations on cholesteryl dimers having mainly hexanoic acid as a spacer [11–16]. They observed that the length of the terminal group and also the nature of the aromatic mesogenic group play a role in the type of smectic ordering that is found. For the dimers with a  $d/l$  ratio of about 1.0, a monolayer type of ordering was suggested. This type of ordering is schematically depicted in figure 7.

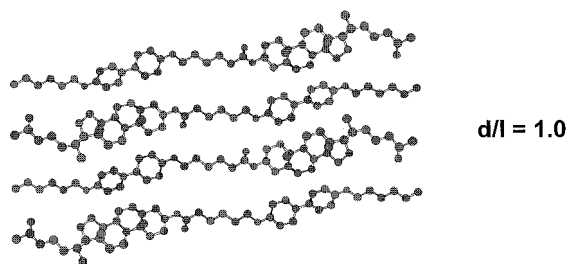


Figure 7. Schematic representation of the possible packing of *mBPnC* molecules with short spacers in the SmA phase (6BP5C in the figure).

The fact that SmA phases are found even for compounds with an odd number of flexible units indicates that the average ordering of the mesogenic groups is perpendicular to the layer plane. This could mean that the average conformation of the spacer allows all the mesogenic groups to be more or less parallel, thus inhibiting tilted phases. Therefore, the perpendicular ordering of the mesogenic groups of the dimers with an odd spacer could be induced by *gauche* conformations in the alkyl spacer. On the other hand, calculations on cholesteryl dimers [14–16] and single crystal XRD on a cholesteryl dimer crystal [21] show that, even in the case of an even spacer, the mesogenic groups are not exactly parallel and actually assume more or less a bow-shape. This indicates that for the description of the type of smectic phase, the parity of the connecting spacer may not be very important. This also follows from experiments in which mixtures of odd and even dimers were investigated [22].

The fact that the ordering of the nematic phase of the compounds with an odd spacer is lower than that of compounds with an even spacer, as seen from the  $\Delta S/R$  values at the N\*–I transition, could explain why some dimers with an odd spacer do not form a smectic phase in the transition area between the two types of smectic ordering. It could also explain why 4BP5C is smectic, whereas 4BP4C and 4BP6C are only N\*, due to the better order parameter arising from the higher proportion of *trans*-conformations in the alkyl spacer of this compound.

For the compounds with a longer spacer, XRD measurements show a  $d/l$  ratio of about 0.5. A possible layer arrangement would most likely consist of a rather random mixture of the two types of mesogenic group, while the spacers link at random the mesogenic groups within different layers (figure 8). Such models have been proposed by Luckhurst *et al.* for non-symmetric dimers. Their relative stability is attributed to favourable specific interactions between the unlike mesogenic groups [7, 22]. In our case the mesogenic groups are also very different, both in electronic properties and in cross-sectional area.

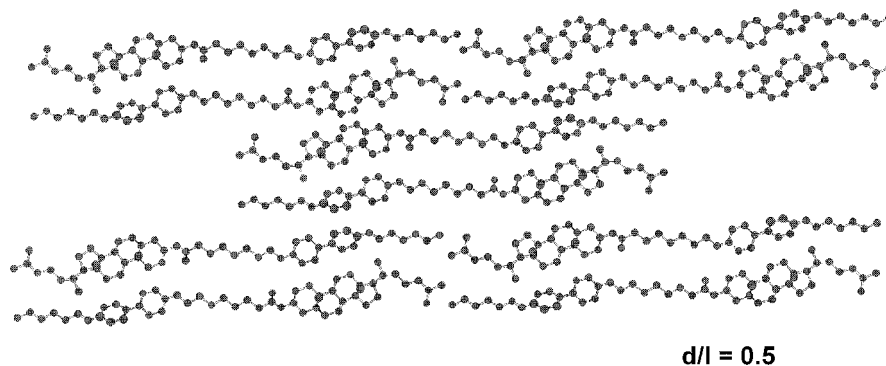


Figure 8. Schematic representation of the possible intercalated packing of *mBPnC* molecules with long spacers in the SmA phase (6BP7C in the figure).

Although these models nicely explain some features, in reality the interactions may be more subtle and governed by three-dimensional steric and electronic interactions. Indications of this come, for example, from the crystal structure of an even cholesteryl dimer, where antiparallel arrangements of both aromatic units and cholesteryl units in a layer are present [21].

From the table it is clearly seen that for the alkoxybiphenyl dimers a small  $\Delta S/R$  change at the SmA–N\* transition corresponds nicely with a  $d/l$  ratio of 1 and a larger  $\Delta S/R$  value with a  $d/l$  ratio of 0.5. For the CN $n$ C compounds with a  $d/l$  ratio of about 1.7, relatively small  $\Delta S/R$  values are also observed. It is interesting to note that the SmA–N\* transitions that have rather low  $\Delta S/R$  values, namely those related to a SmA phase with a  $d/l$  ratio of 1.0 and  $\geq 1.7$ , are accompanied by an intermediate TGB phase, whereas those with a large  $\Delta S/R$  value and related to an intercalated SmA phase with a  $d/l$  ratio of 0.5, do not show an intermediate TGB phase. This phenomenon is not restricted to cholesterol-containing dimers, because the same observations with respect to the relation between  $d/l$  ratio and the presence of a TGB phase were made for non-cholesterol-containing chiral dimers [6]. In this study it was stated that the intercalated nature of the SmA phase might well inhibit the layers from twisting into a helix.

From figure 3 it is seen that for small  $n$  the entropy change first decreases with  $n$ . This could indicate that the monolayer ordering destabilizes with increasing  $n$ . The entropy change of the SmA<sub>c</sub>–N\* transition seems to reach a maximum at  $n = 7$ , before it decreases again for  $n = 10$ . This may indicate that an optimal ordering in the SmA phase with a  $d/l$  ratio of about 1 is obtained for  $n \leq 3$  and in the SmA phase with a  $d/l$  ratio of about 0.5 for  $n \approx 7$ –8.

#### 4. Conclusions

Three series of dimer LCs containing a cholesteryl and an alkoxybiphenyl group were investigated and show two modifications of the SmA phase. For short spacers, a SmA phase is found with a  $d/l$  ratio of about

1. In these cases the transition entropy ( $\Delta S/R$ ) from SmA to N\* is small. For longer spacers, a SmA phase is found with a  $d/l$  ratio of about 0.5 and the transition entropy from SmA to N\* is larger. With shorter terminal groups, the change in smectic properties shifts to a smaller spacer length. In every series there are one or two compounds that exhibit only a N\* phase and no SmA phase. This failure to give a SmA phase was observed only for dimers with an odd number of flexible units in their spacer and when spacer and terminal group have about equal lengths. For a series of dimer LCs containing a cholesteryl and a cyanobiphenyl group the smectic behaviour is completely different. For this series, an interdigitated smectic layer spacing is found that is about 1.7 times the length of the molecules.

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