Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713926090

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To cite this Article Lee, Jun-woo , Jin, Jung-il , Achard, M. F. and Hardouin, F.(2003) 'Cholesterol-based hydrogen-bonded liquid crystals', Liquid Crystals, 30: 10, 1193 — 1199

To link to this Article: DOI: 10.1080/02678290310001599233

URL: http://dx.doi.org/10.1080/02678290310001599233

LIQUID CRYSTALS, VOL. 30, NO. 10, OCTOBER, 2003, 1193–1199

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Cholesterol-based hydrogen-bonded liquid crystals

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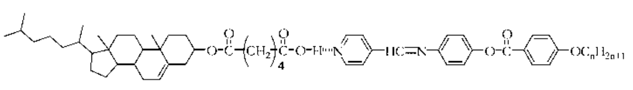
(*Received 31 January 2003; in final form 14 April 2003; accepted 13 May 2003*)

Hydrogen bonding is a powerful tool for assembling molecules and building new liquid crystalline structures. In this study, non-symmetric dimesogens were prepared by intermolecular hydrogen bonding between rationally designed H-bond donor (3-cholesteryloxycarbonylpentanoic acid) and acceptor (4-(pyridine-4-ylmethyleneimino)phenyl 4-alkoxybenzoate) moieties. Their liquid crystalline properties were investigated by differential scanning calorimetry, polarized optical microscopy and X-ray diffraction. Cholesteric and smectic phases were observed. As for the covalently linked dimesogens, several types of smectic periodicities occur for these H-bonded cholesteryl compounds depending on the molecular parameters.

1. Introduction

In recent years, there has been much interest in liquid crystalline (LC) twin, dimeric or dimesogenic compounds that contain two identical [1–5] or different [5–16] mesogenic units. The role of hydrogen bonding interactions in the formation and/or stabilization of liquid crystalline phases has been extensively studied over the last 10 years. The first hydrogen-bonded complexes exhibiting liquid crystalline behaviour resulted from the dimerization of carboxylic acids [17]. The concept of building mesogenic structures through hydrogen bonding between H-bond donors and acceptors has been extended to a variety of mesogenic compounds and, depending on the number and position of the groups able to form hydrogen bonds, a large diversity of supramolecular structures has been reported and recently reviewed [18]. Kato and co-workers [19–22] showed that hydrogen bonding between carboxyl and pyridyl moieties, among the possible range of interacting components, is very useful for the formation of supramolecular hydrogen-bonded liquid crystals. Many materials, both dimeric and polymeric, derived from these moieties have been synthesized [18].

The objective of the present work was to synthesize through H-bonding [23, 24] new cholesterol-based dimesogenic compounds (**CH6A-SEOC*n***. $n = 4$–8 and 10) which exhibit smectic mesophases.



CH6A-SEOC*n* (n =4~8 and 10)

In a preliminary report [24] we have described the LC properties of **CH6A-SEOC4** and **CH6A-SEOC10**. Dimesogens composed of a conventional rod-like mesogen covalently linked to a cholesteryl moiety by an aliphatic spacer show rather intriguing smectic phase behaviour: specifically, they form smectic phases with different types of lamellar packing and, depending on the molecular parameters, the layer spacing can be smaller than one half the molecular length or, alternatively, larger than the molecular length. More unusually, between these two extremes, anomalies of periodicity are revealed through the occurrence of two-dimensional modulated phases. In some rare cases, an incommensurate fluid smectic phase (Sm$_{\text{inc}}$) is observed in which the two competing periodicities coexist in the long spacing range [11–16]. In this study, we prepared similar dimesogens involving H-bonding in order to compare their mesomorphic and structural properties with those of the corresponding, covalently linked dimesogens. In order to obtain smectic phases for the complexes induced by hydrogen bonding, we selected

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Liquid Crystals ISSN 0267-8292 print/ISSN 1366-5855 online
http://www.tandf.co.uk/journals
DOI: 10.1080/02678290310001599233

proton donors and proton acceptors which themselves exhibit liquid crystalline phases.

2. Experimental

2.1. *Characterization*

The IR and ^{1}H NMR spectra were recorded on a Bomem MB FTIR spectrophotometer and Bruker AM300 spectrometer, respectively. The thermal transition temperatures and optical textures of all the products were obtained using a Perkin-Elmer DSC7 differential scanning calorimeter and a Leitz Diavert polarizing microscope equipped with a heating stage (Mettler FP-82HT) and an automatic controller (Mettler FP-90), respectively. The Heating and cooling rate was 5°C min^{-1}. X-ray diffraction (XRD) patterns of powder and aligned samples were obtained using CuK$_\alpha$ radiation from an 18 kW rotating anode X-ray generator (Rigaku-200) selected by a flat (1 1 1) germanium monochromator. The scattered radiation was collected on a two-dimensional detector (Imaging Plate system from Mar Research, Hamburg, Germany). The samples were placed in an oven on the X-ray apparetus, providing a temperature control of 0.1 K.

2.2. *Synthesis*

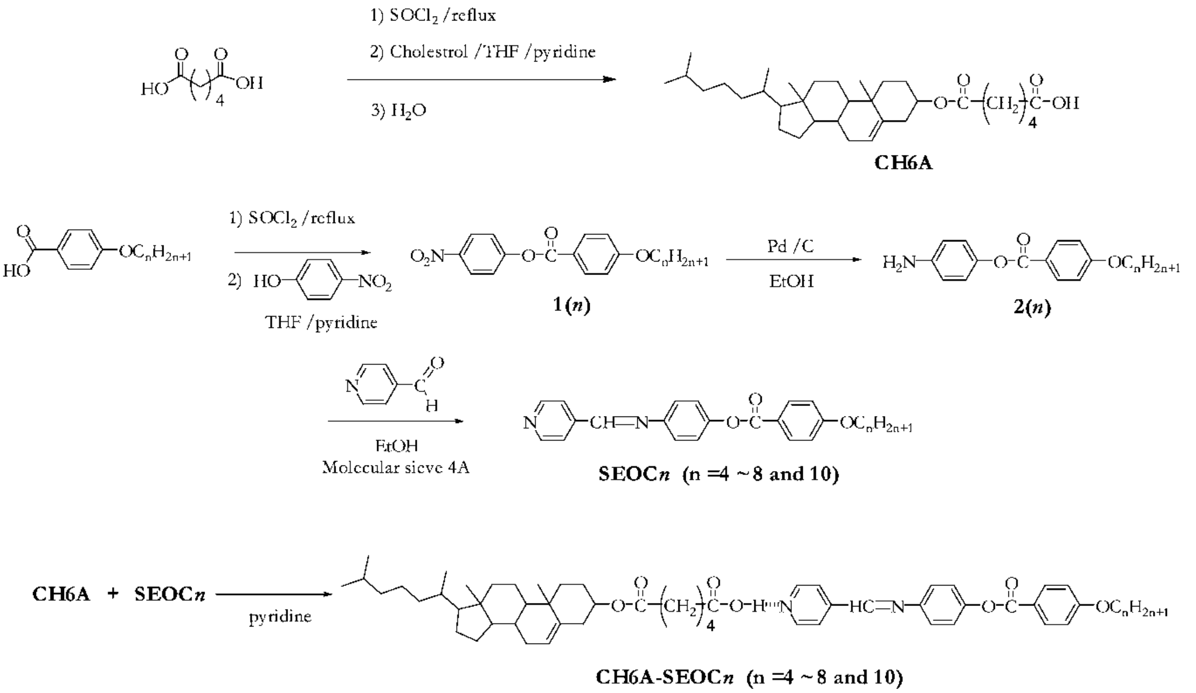
The synthetic route to these compounds is shown in the scheme, and is essentially the same as we have earlier reported [24] for similar homologous series of dimesogenic compounds. Therefore, here we give only representative spectral and elemental analysis data for the hydrogen bond acceptors and hydrogen bond complexes.

4-[(Pyridine-4-ylmethylene)amino]phenyl 4-pentyloxybenzoate, SEOC5. Yield 1.9 g (47%), m.p. 113°C. FTIR (KBr, cm^{-1}): 3054 (aromatic C–H stretching), 2953 (aliphatic C–H stretching), 1721 (C–O stretching), 1605 and 1507 (aromatic C–C stretching), 1258 and 1068 (C–O stretching). ^{1}H NMR (CDCl$_3$, ppm): δ 8.8 (d, 2H), 8.1 (d, 2H), 7.7 (d, 2H) 7.3 (m, 4H) and 7.0 (d, 2H, aromatic Hs), 8.5 (s, –N–$\underline{CH}$–), 4.0 (t, 2H, –PhO$\underline{CH_2}$–), 1.8 (m, 2H, –OCH$_2\underline{CH_2}$–), 1.5 (m, 4H, OCH$_2$CH$_2\underline{CH_2CH_2}$–), 1.0 (3H, m, –$\underline{CH_3}$). Elemental analysis: calc. for $C_{24}H_{24}N_2O_3$ C 74.21, H 6.23, N 7.21; found C 74.15, H 6.31, N 7.20%.

CH6A-SEOC5 complex. FTIR (KBr, cm^{-1}): 2944 and 2870 (aliphatic C–H stretching), 2648–2230 and 2048–1799 (hydrogen bond O–H stretching), 1728 (C–O stretching), 1606 and 1506 (aromatic C–C stretching), 1255 and 1071 (C–O stretching). ^{1}H NMR (CDCl$_3$, ppm): δ 8.8 (d, 2H), 8.1 (d, 2H) 7.8 (d, 2H), 7.3 (m, 4H) and 7.0 (d, 2H aromatic Hs), 8.5 (s, –N–$\underline{CH}$–), 5.4 (m, 1H, –C–$\underline{CH}$–), 4.6 (m, 1H, –(CH$_2$)$_2\underline{CH}$–O–), 4.0 (t, 2H, –PhO$\underline{CH_2}$–), 2.4–2.5 (m, 2H, –$\underline{CH_2}$–COOH) , 0.6–2.1 (m, 56H, –CH–, –CH$_2$– and –CH$_3$). Elemental analysis: calc. for $C_{57}H_{78}N_2O_7$ C 75.80, H 8.70, N 3.10; found C 75.73, H 8.77, N 3.11%.

3. Results and discussion

The thermal behaviour of the H-bond donor and acceptors and of the 1:1 complexes was examined by

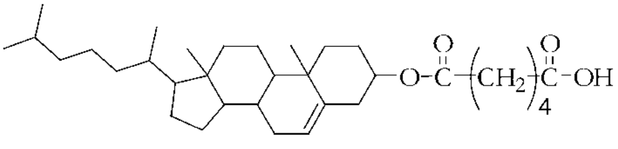


Scheme. Synthetic route used to obtain the **CH6A-SEOC*n*** complexes.

DSC, and an initial characterization of the liquid crystalline phases was carried out using polarizing optical microscopy. XRD experiments were performed to obtain structural identification.

3.1. *Mesomorphic properties*

3-Cholesteryloxycarbonylpentanoic acid (**CH6A**) was chosen as the proton donor:



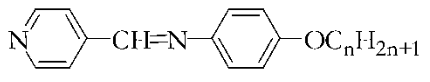
CH6A

We found that this compound shows an enantiotropic cholesteric phase between 134 and 148°C and exhibits classic oily streak or fan-shaped textures. By comparison, **CH4A** (two methylene groups in the dicarboxylic acid moiety, melting temperature 178.5°C) is not mesogenic, while **CH5A** (three methylene groups, melting temperature 112°C) forms the cholesteric phase only monotropically.

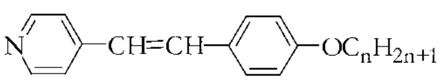
Initially, we used molecules with two phenyl ring, such as *n*-(4-pyridylmethylidene)alkoxyanilines (**SchiffOC*n***) or 4-alkoxystilbazoles (**SbOC*n***) as proton acceptors. These compounds are not mesogenic. Nevertheless, the 1:1 complexes prepared with **CH6A** exhibited cholesteric mesophase, but only monotropic smectic phases were observed. Some examples are given in table 1.

Table 1. Transition temperatures (obtained by DSC) of the 1:1 complexes obtained using **CH6A** and *n*-(4-pyridylmethylidiene)alkoxyanilines (**SchiffOC*n***) and 4-alkoxystilbazoles (**SbOC*n***). Cr: crystalline phase, N* = cholesteric, Sm = smectic, [ ] indicates a monotropic transition.

Compound	Transition temperature °C				
CH6A-SchiffOC4	Cr	113	N*	137	I
CH6A-SchiffOC10	Cr	100	N*	127	I
	[Sm]	99	↲		
CH6A-SbOC3	Cr	126	N*	151	I
	[Sm]	107	↲		
CH6A-SbOC4	Cr	128	N*	150	I
	[Sm]	111	↲		
CH6A-SbOC5	Cr	117	N*	147	I
	[Sm]	101	↲		



SchiffOC*n* (*n* =4 and 10)



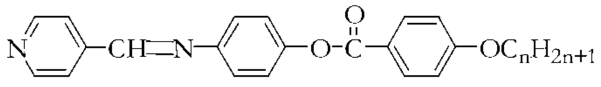
SbOC*n* (*n* =3~5)

Thus, in order to facilitate the formation of smectic phases in the H-bonded supramolecular structures, we decided to use mesogens with three phenyl rings as the proton acceptors. As shown in table 2, the 4-(pyridine-4–ylmethyleneimino)phenyl 4-alkoxybenzoates (**SEOC*n***) all are mesomorphic.

Table 2. Transition temperatures and enthalpies (in parentheses) (obtained by DSC) for the H-bond donor and acceptors. Cr = crystalline phase, SmA = smectic A, SmC = smectic C, N = nematic, [ ] indicates a monotropic transition.

Compound	Transition temperatures °C; enthalpy changes kJ mol^{-1}								
CH6A[a]		(24.5)		(1.5)					
	Cr	134	N*	148	I				
SEOC4[a]		(40.9)		(0.4)					
	Cr	138.5	N	160	I				
SEOC5		(24.7)		(0.2)					
	Cr	113	N	158.1	I				
SEOC6		(27.2)		(0.2)					
	Cr	111.2	N	147.2	I				
	[SmA]	107	↲(0.12)						
SEOC7		(28.8)		(0.22)		(0.3)			
	Cr	111	SmA	117.8	N	141.8	I		
SEOC8		(26.7)		(0.17)		(0.4)			
	Cr	99	SmA	125.5	N	152.3	I		
SEOC10[a]		(40.9)		(-)		(1.6)		(0.8)	
	Cr	94	SmC	133	SmA	143	N	145	I

[a] From [24]



SEOC*n* (*n* =4~8 and 10)

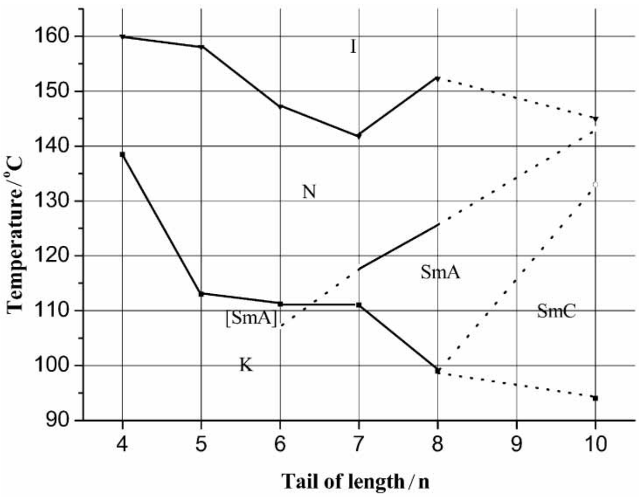


Figure 1. Transition temperatures as a function of chain length for the **SEOC*n*** series. SmA = smectic A, SmC = smectic C, N* = cholesteric, I = isotropic liquid, [SmA] = monotropic smectic A.

The first members of the series are only nematic. The longer homologues (*n* = 6–8 and 10) show a nematic (N) – smectic A (SmA) sequence and the compound **SEOC10** an additional smectic C (SmC) mesophase.

The transition temperatures of the series are shown in figure 1. In general, the melting temperature is reduced as the length of the alkyloxy group increases, whereas the SmA – N transition temperature increases with the chain length. Evidently, the longer chains tend to stabilize the SmA phase, most probably due to enhanced interactions between the chain ends [25–27]. The clearing temperature is initially lowered and then increases with chain length. **SEOC10** exhibits a rather broad SmC phase range (*c.* 40°C) and a very short SmA phase, of 2°C.

The 1:1 complexes are prepared from an equimolar mixture of 3-cholesteryloxycarbonyl pentanoic acid (**CH6A**) as the proton donor and a 4-(pyridine-4-ylmethyleneimino)phenyl 4-alkoxybenzoate (**SEOC*n*** with *n* = 4–8 and 10) as the proton acceptor. The **CH6A-SEOC*n*** complexes thus prepared behave as single component liquid crystalline materials and exhibit stable mesophases. As shown in table 3, the clearing temperature increases noticeably for the 1:1 complexes in comparison with those of the precursors. In addition, the mesomorphic range is clearly extended due to the formation of supramolecular liquid crystals. All the complexes under study display a cholesteric mesophase. Upon further cooling, a SmA phase is observed for *n* = 4–8. For *n* = 8, a chiral smectic C (SmC*) phase occurs just before crystallization. The decyloxy homologue only exhibits a cholesteric – SmC* sequence. The **CH6A-SEOC4** complex displays an additional fluid smectic phase identified by X-ray analysis to be an incommensurate smectic A phase [24]. Figure 2 illustrates the dependence of phase behaviour on the chain length of the **SEOC*n*** moiety for these complexes.

Table 3. Transition temperatures and associated enthalpies (obtained by DSC) for the 1:1 **CH6A-SEOC*n*** complexes. Cr = crystalline phase, SmA = smectic A, $\mathrm{SmA_{inc}}$ = incommensurate smectic A (from XRD), SmC* = chiral smectic C, N* = cholesteric, I = isotropic liquid, [ ] indicates a monotropic transition. (-) very small enthalpy value.

Compound	Transition temperature °C; enthalpy kJ mol^{-1}						
CH6A-SEOC4[a]		(40.8)		(0.18)		(3.95)	
	Cr	126	SmA	145	N*	192	I
	[$\mathrm{SmA_{inc}}$]	116	↲(0.18)				
CH6A-SEOC5		(34.9)		(0.22)		(3.4)	
	Cr	125.4	SmA	156.2	N*	189.7	I
CH6A-SEOC6		(45.7)		(0.23)		(4.1)	
	Cr	130.9	SmA	153.6	N*	188.3	I
CH6A-SEOC7		(46.8)		(0.15)		(3.20)	
	Cr	124	SmA	149	N*	184.1	I
CH6A-SEOC8		(37)		(-)		(4.0)	
	Cr	125	SmA	145	N*	183.6	I
	[SmC*]	111	↲(0.18)				
CH6A-SEOC10[a]		(38.7)		(0.25)		(5.6)	
	Cr	126	SmC*	135	N*	192	I

[a]Reported [24]

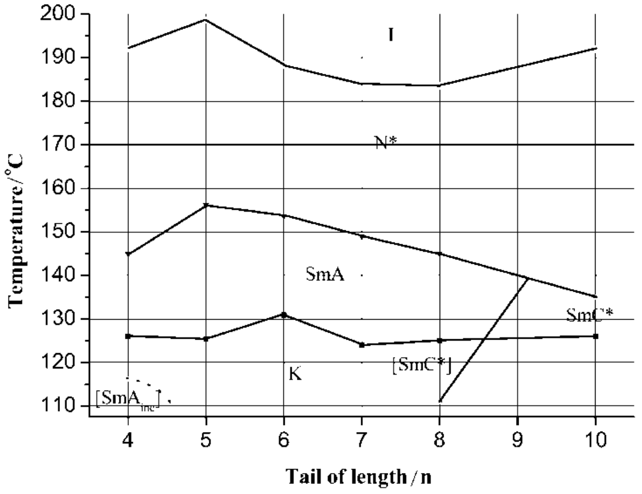


Figure 2. Dependence of the transition temperatures on the chain length in the H-bond acceptor for the **CH6A-SEOC*n*** complexes. SmA = smectic A, SmA$_{inc}$ = incommensurate smectic A, SmC* = chiral smectic C, N* = cholesteric, I = isotropic liquid.

3.2. *Structural analysis by X-ray diffractometry*

The X-ray patterns of the smectic phases of both the **SEOC*n*** precursors and the **CH6A-SEOC*n*** complexes contain Bragg peaks in the low angle region corresponding to the layer reflections and diffuse scattering at wide angle indicating a liquid-like order within the layers.

For **SEOC*n*** (n = 7, 8 and 10), the layer spacing d is constant in the SmA phase (table 4), and decreases from 35 to 33.8 Å with decreasing temperature in the SmC phase of **SEOC10** (table 4) indicating an increase of the tilt angle. The SmA phase is partially bilayered, since the layer spacing (29–35 Å) is larger than the molecular length (27.5–31.4 Å) in the most extended conformation. For the SmA phase of the butyloxy (n = 4; **CH6A-SEOC4**) to octyloxy (n = 8; **CH6A-SEOC8**) complexes, the intensity profile in the low angle region displays a Bragg peak corresponding to a layer spacing shorter than half the complex length. The layer periodicity lies between 23.6 and 25.7 Å and, as previously observed in covalently linked dimesogens, this quasi constant value (*c.* 24–26 Å) is imposed by the cholesteryl moiety. The estimated molecular length was estimated using Chem3D software assuming that all the methylene groups in the complexes are in a fully extended *trans* zig-zag conformation and that hydrogen-bonded pyridinium carboxylate complexes form. The values included in table 4 show that the molecular length increases from 48.7 to 56.1 Å as we increase the chain length from butoxy to decyloxy.

In all the X-ray patterns an additional diffuse scattering is observed. It corresponds to smectic fluctuations connected to the full complex length. This diffuse scattering becomes increasingly more visible as the temperature decreases in the mesophase. As shown in table 4, these fluctuations, shown in brackets, correspond to an additional smectic periodicity, which only develops in the low angle region and decreases from 60.4 to 51.1 Å on increasing the alkoxy terminal length. In the case of **CH6A-SEOC4**, the long spacing in the SmA phase estimated from the XRD pattern is significantly larger than the estimated molecular length, 60.4 Å vs. 48.7 Å. The difference between the two values is reduced on increasing the chain length and finally vanishes for **CH6A-SEOC6**, 51.0 Å vs. 51.2 Å. The long spacing observed then becomes smaller than the theoretical molecular length as we further increase the chain length.

Table 4. Structural data for the **SEOC*n*** precursors and the **CH6A-SCEOC*n*** complexes. < > indicates a diffuse scattering and not a Bragg reflection.

Compounds	SmA phase layer spacing Å	Sm C or SmC* phase layer spacing Å	Estimated molecular length/Å
SEOC7	21.9		27.5
SEOC8	30.1		28.7
SEOC10[a]	35.1	35–33.8 (decreasing temperature)	31.4
CH6A-SEOC4[a]	In SmA d = 23.6 + < d = 60.4 > In SmA$_{inc}$ d = 23.6 and d = 60.4		48.7
CH6A-SEOC5	d = 24.25, fluctuations at < d = 52.3 >		50.1
CH6A-SEOC6	d = 24.7, fluctuations at < d = 51.0 >		51.2
CH6A-SEOC7	d = 25.2, fluctuations at < d = 50.2 >		52.4
CH6A-SEOC8	d = 25.7, fluctuations at < d = 51.1 >	Crystallisation prevents study in the SmC* phase	53.7
CH6A-SEOC10[a]		52–45 (decreasing temperature)	56.1

[a] Reported [24]

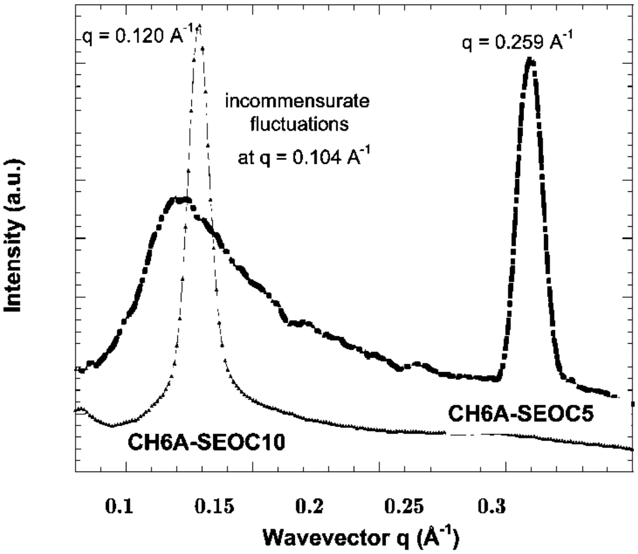


Figure 3. Intensity profiles for two H-bonded complexes with different aliphatic chain lengths $n =$ for a short chain (**CH6A-SCEOC5**) the Bragg peak observed at 0.259 Å^{-1} corresponds to a layer spacing of 24.2 Å and the intense diffuse scattering centred around 0.120 Å^{-1} reveals incommensurate fluctuations (*i.e.* not a Bragg reflection) corresponding to 52 Å. For the long chain complex (**CH6A-SCEOC10**) an inversion of the smectic periodicity is observed with a layer spacing of 49 Å at127°C.

For the short chain members, these fluctuations clearly correspond to incommensurate fluctuations (incommensurability ratio: 60.4/23.6 = 2.56 for $n = 4$, 2.16 for $n = 5$ and 2.06 for $n = 7$) [11,12]. This tendency, which is more pronounced for the butyloxy **CH6A-SEOC4** [8] complex, favours the occurrence of an incommensurate smectic A (SmA$_{\text{inc}}$) phase at lower temperature; the signature of this phase is the sharpening of the diffuse scattering into a Bragg reflection and the simultaneous existence of two incommensurate peaks at $q = 0.104$ Å^{-1} (60.4 Å) and $q = 0.266$ Å^{-1} (23.6 Å). An oriented pattern confirmed that the two incommensurate wave vectors are collinear [13] and this indicates the existence of an incommensurate fluid smectic SmA$_{\text{inc}}$ for this H-bonded dimesogen. This is the first example of a H-bonded dimesogen which exhibits an incommensurate smectic A phase.

Increasing the chain length favours a commensurate lock-in and the larger periodicity prevails. Thus, for the smectic C* phase of the decyloxy complex, i.e. **CH6A-SEOC10** [24], a sudden change in the smectic layering is observed (see figure 3). This parameter now corresponds to a smaller value (52 Å) than the complex length (56.1 Å). This periodicity decreases from 52 to 45 Å as the temperature is lowered from 135 to 126°C. Thus, as in covalently linked dimesogens, two competing periodicities exist in H-bonded cholesteryl dimesogens: one periodicity is larger than (or close to) the complex length and the other smaller than (or close to) half the complex length. We observe also the same inversion of the smectic periodicity on increasing the terminal alkoxy chain length. The observation of an incommensurate smectic A phase reveals a competition between two incommensurate periodicities in the **CH6A-SEOC*n*** complexes as previously observed for the covalently linked dimesogen [11–13]. The results presented here demonstrate that the H-bonded dimesogens behave as single components with properties comparable to those of covalently linked dimesogens.

This collaborative research was co-sponsored by the Korea Science and Engineering Foundation through the Center for Electro- and Photo-Responsive Molecules, Korea University and by the CNRS of France through the Paul-Pascal Research Center, Pessac, France.

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