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Synthesis and mesomorphic properties of a novel series of cholesterol-based liquid crystalline tetramers

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A new series of symmetric liquid crystal tetramers involving cholesteryl-based mesogenic units and Schiff's base moiety were designed and synthesised. The target compounds were obtained by the reaction of 4-(ω-cholesteryloxycarbonylalkonoyloxy) benzaldehyde with compound 1,4-bis (aminophenyl-1-oxy) butane. The length of the outer two spacers is varied from 2 to 8 even-numbered methylene units, while the central spacer is held at 4 methylene units. The molecular structures of the intermediates and target compounds were confirmed by Fourier transform infrared and proton nuclear magnetic resonance spectroscopy. The thermal phase behaviour of the synthesised tetramers were investigated by polarising optical microscopy coupled with hot stage and differential scanning calorimetry. All of these liquid crystal tetramers showed only one chiral nematic mesophase on a very large temperature domain (~100°C), and the clearing temperature above 250°C.

Keywords: cholesterol-based; Schiff's base; liquid crystalline tetramers; synthesis

1. Introduction

Over the last decade, cholesterol-based liquid crystals have attracted attention from more and more researchers, not only due to the fact that cholesterol is widespread in nature and commercially available, but also because the helical supermolecular structure of cholesterol-based liquid crystals imparts some special optical properties, including optical rotation, circular dichroism and selective reflection. Additionally, these properties are extremely sensitive to external conditions such as temperature, pressure and electric field, and thus they could be potential candidates for application in optical storage, colour display techniques and full colour rewritable recording devices (*1–3*).

Imrie *et al.* have published some critical reviews (*4–6*), which systematically describe the relationships between molecular structure and mesomorphic behaviour in liquid crystal oligomers, and in particular in dimers. To date, some liquid crystal dimers (*7–23*) and trimers (*24–27*) consisting of a cholesteryl ester unit as a chiral segment joined to different mesogenic moieties such as a benzoate ester, Schiff's base, azobenzene, biphenyl or tolane, etc., through flexible spacers, have been synthesised and extensively studied. They have shown rich and interesting mesomorphic phase behaviour including frustrated SmA phases, TGB phases, blue phases, and the chiral nematic (N*) mesophase. Their liquid crystalline (LC) properties are dependent on the length and parity of the spacers, the terminal group attached to the aromatic rings and the types of bridging group between the two aromatic rings. This research forms the foundations now for further investigations on the relationship between structure and property in cholesterol-based oligomers.

With the aim of better understanding the structure–property correlations in liquid crystal oligomers and to investigate how the mesomorrphic properties evolve from dimers to polymers, some tetramers have been prepared and characterised (*28–33*). The first complete homologous series of linear liquid crystal tetramers was reported by Imrie *et al.* (*28*). They are composed of biphenyl mesogenic entities and Schiff's base moieties linked through alkyl spacers (n = 3–12). This series exhibited nematic and smectic mesophases. The clearing temperatures and associated entropy changes of these liquid crystal tetramers show a dramatic odd–even effect just like the dimers and trimers. Compared with conventional low molar mass mesogens, these compounds have considerably higher clearing entropies. However, reports describing the synthesis of tetramers bearing a cholesteryl unit are relatively few. Yelamaggad *et al.* designed and synthesised the first tetramers possessing four non-identical mesogenic units, namely, tolane, azobenzene, biphenyl and cholesteryl ester (*32*). These linear tetramers displayed a columnar mesophase and were found to bridge the gap between low molar mass liquid crystals and polymers, mainly because they can simultaneously exhibit properties associated with polymer and low molar mass liquid crystals.

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In light of these observations and the fact that no reports to date have described dicholesterol-based tetramesogenic compounds, we aimed to design and prepare a series of novel liquid crystal tetramers incorporating dicholesteryl units and two Schiff's base moieties and study their influence on liquid crystal properties by changing the length of the outer two spacers. We will see that this series of compounds exhibits solely chiral nematic mesophase behaviour over a wide range of temperatures.

2. Experimental details

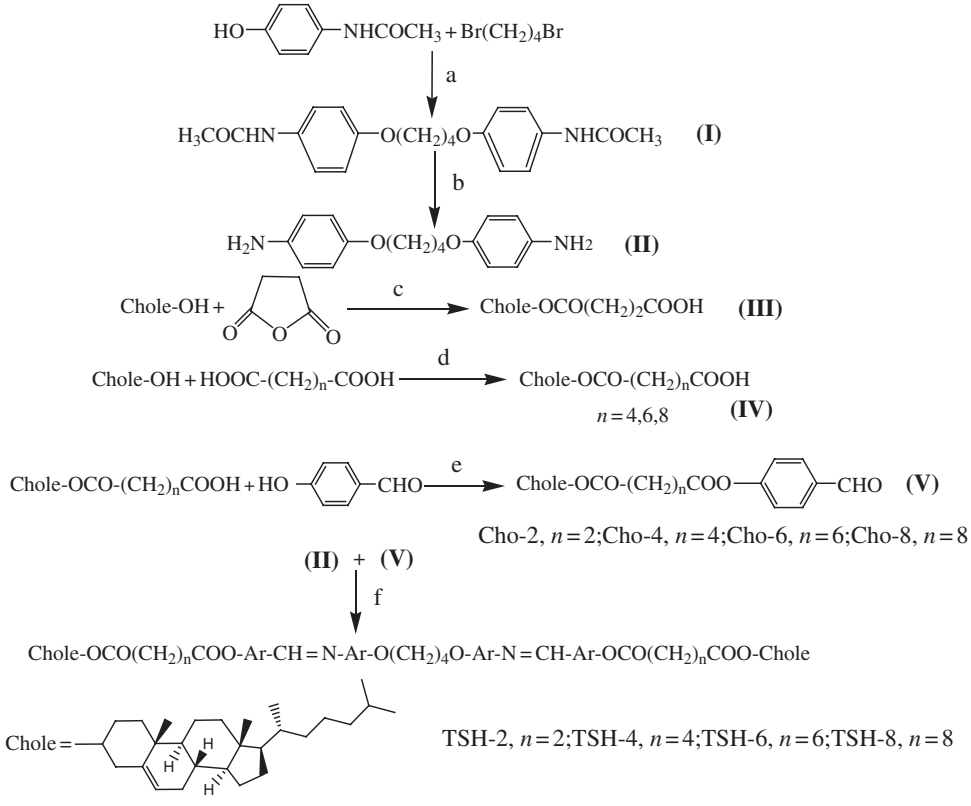
2.1 Characterisation

IR spectra were recorded on an FT-IR Bruker Tensor 307 spectrometer by using KBr pellets, and proton nuclear magnetic resonance (^{1}H NMR) spectra were measured with a Bruker 300 MHz instrument in $CDCl_3$ with tetramethylsilane (TMS) as internal standard. The liquid crystalline textures were observed by means of an Olympus BX51 polarising optical microscope (POM) equipped with a Linkam THMS600 hot stage. Differential scanning calorimetry (DSC; Perkin-Elmer DSC-7) was carried out to measure the transformation temperature and associated enthalpy values at a scanning rate of 5°C min^{-1} during the heating process.

2.2 Synthesis

Cholesterol, as a biochemical reagent, was obtained from Tianjin Yingbo Biochemical Reagent Company (Tianjin, China); N,N-dicylcohexylcarbodiimide (DCC) and 4-dimethylaminopyridine (DMAP) were used as received (Arcos). Tetrahydrofuran (THF) was distilled from sodium–benzophenone; dichloromethane (DCM) was refluxed over CaH_2 and distilled prior to use. All other solvents and reagents were AR or CP grade and used without further purification. The synthetic route used to prepare this series of tetramers is depicted in Scheme 1.

Compound1,4-bis(acetaminophenyl-1-oxy)butane (I) and 1,4-bis(aminophenyl-1-oxy)butane (II) was synthesised as previously described in the literature (*34*).The starting compounds 3-cholesteryloxycarbonyl propanoic acid (III) and dicarboxylic acid monocholesteryl ester (IV) were synthesised according to a reported procedure (*35*).



Scheme 1. Synthesis route of the liquid crystal tetramers. Reagents and conditions: (a) K_2CO_3, acetone, reflux; (b) i, Concentrated HCl, reflux; ii, NaOH; (c) pyridine/heptane, 21 h; (d) DCC/DMAP/THF, 24 h; (e) DCC/DMAP/DCM, room temperature, 24 h; (f) absolute ethanol/p-toluene sulphonic acid, reflux.

2.2.1 Synthesis of 4-(3-cholesteryloxycarbonyl propionyloxy) benzaldehyde (Cho-2)

1.948 g (4 mmol) succinic acid monocholesteryl ester and 0.488 g (4 mmol) p-hydroxy benzaldehyde were dissolved in 40 ml dry DCM. 0.824 g (4 mmol) DCC and a catalytic amount of DMAP were then added with stirring at room temperature for 24 h. The dicyclohexylurea formed was removed by filtration and the filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (100–200 mesh) using a mixture of petroleum ether (60–90°C) and ethyl acetate (4/1, v/v) as eluent, and then recrystallised from ethanol to yield white powder. Yield: 75%. Cr104.1Sm126.3Ch134.8I.

Fourier transform infrared (FTIR) (KBr, cm^{-1}): 2940, 2737, 1766, 1736, 1707, 1599, 1310, 1126, 979, 857.

^{1}H NMR ($CDCl_3$, ppm): 9.99 (s,1H), 7.90 (t, 2H), 7.26 (t, 2H), 5.37 (m,1H), 4.65 (m, 1H), 2.90 (t, 2H), 2.73 (t, 2H), 2.35–0.68 (broad, 43H).

The procedures for the preparation of 4-(5-cholesteryloxycarbonyl pentanoyloxy) benzaldehyde (Cho-4), 4-(7-cholesteryloxycarbonyl heptanoyloxy)benzaldehyde (Cho-6) and 4-(9-cholesteryloxycarbonyl nonanoyloxy) benzaldehyde (Cho-8) were similar to the procedure described above.

Cho-4: White powder (yield: 61%). Cr123.8Ch143.5I.

FTIR (KBr, cm^{-1}): 2924, 2735, 1772, 1705, 1735, 1598, 1250, 1157, 1004, 855.

^{1}H NMR ($CDCl_3$, ppm): 9.99 (s, 1H), 7.95 (t, 2H), 7.30 (t, 2H), 5.38 (m, 1H), 4.67 (m, 1H), 2.64 (m, 4H), 2.37–0.69 (broad, 47H).

Cho-6: White powder (yield: 60%). Cr127.7Ch136.8I.

FTIR (KBr, cm^{-1}): 2939, 2719, 1767, 1731, 1700, 1593, 1291, 1173, 1009, 840.

^{1}H NMR ($CDCl_3$, ppm): 10.02 (s, 1H), 7.94 (t, 2H), 7.24 (t, 2H), 5.39 (m, 1H), 4.64 (m, 1H), 2.61 (m, 4H), 2.34–0.70 (broad, 51H).

Cho-8: White powder (yield: 61%). Cr98.6Ch111.7I.

FTIR (KBr, cm^{-1}): 2929, 2714, 1762, 1726, 1700, 1598, 1296, 1152, 1009, 860.

^{1}H NMR ($CDCl_3$, ppm): 10.02 (s, 1H), 7.94 (t, 2H), 7.31 (t, 2H), 5.40 (m, 1H), 4.65 (m, 1H), 2.60 (m, 4H), 2.35–0.70 (broad, 55H).

2.2.2 Cholesterol-based tetramesogenic compounds (TSH-2, TSH-4, TSH-6 and TSH-8)

The cholesterol-based tetramesogenic compounds were synthesised following a common general synthetic procedure. 1 mmol compound Cho-*n* (*n* = 2, 4, 6, 8) and 0.5 mmol 1,4-bis (4-aminophenyl-1-oxy) butane were dissolved in 40 ml absolute ethanol, and the mixture was refluxed in the presence of traces of p-toluene sulphonic acid (PTSA) as catalyst for 4 h. After cooling to room temperature, the resulting precipitate was filtered off, washed with ethanol several times and dried in vacuum. The solid product TSH-*n* (*n* = 2, 4, 6, 8) was obtained.

TSH-2: White solid powder (yield: 82%).

FTIR (KBr, cm^{-1}): 2945, 1760, 1731, 1623, 1576, 1506, 1311, 1131, 978, 949, 834.

^{1}H NMR ($CDCl_3$, ppm): 8.46 (s, 2H), 7.90 (d, 4H), 7.22 (d, 8H), 6.93 (d, 4H), 5.39 (m, 2H), 4.66 (m, 2H), 4.08 (t, 4H), 2.90 (t, 4H), 2.73 (t, 4H), 2.34 (d, 4H), 2.04–0.68 (broad, 86H).

TSH-4: White solid powder (yield: 68%).

FTIR (KBr, cm^{-1}): 2939, 1762, 1731, 1625, 1598, 1506, 1255, 1157, 1013, 922, 840.

^{1}H NMR ($CDCl_3$, ppm): 8.46 (s, 2H), 7.93 (d, 4H), 7.25 (d, 8H), 6.92 (d,4H), 5.38 (m,2H), 4.63 (m,2H), 4.07 (t,4H), 2.61 (m,8H), 2.34 (m,4H), 2.01–0.67 (broad,94H).

TSH-6: White solid powder (yield: 65%).

FTIR (KBr, cm^{-1}): 2945, 1751, 1731, 1625, 1603, 1506, 1234, 1157, 1014, 922, 835.

^{1}H NMR ($CDCl_3$, ppm): 8.46 (s, 2H), 7.94 (d, 4H), 7.18 (d, 8H), 6.93 (d, 4H), 5.38 (m, 2H), 4.64 (m, 2H), 4.08 (t, 4H), 2.58 (m, 8H), 2.31 (m, 4H), 2.01–0.68 (broad, 102H)

TSH-8: White solid powder (yield: 66%).

FTIR (KBr, cm^{-1}): 2929, 1757, 1736, 1627, 1603, 1511, 1239, 1163, 1009, 922, 835.

^{1}H NMR ($CDCl_3$, ppm): 8.46 (s, 2H), 7.92 (d, 4H), 7.22 (d, 8H), 6.93 (d, 4H), 5.37 (m, 2H), 4.62 (m, 2H), 4.07 (t, 4H), 2.58 (m, 8H), 2.29 (m, 4H), 2.01–0.67 (broad, 110H).

3. Results and discussion

3.1 Synthesis

These new tetramesogens were synthesised according to Scheme 1. The requisite intermediates 4-(ω-cholesteryloxycarbonylalkonoyloxy) benzaldehyde (Cho-*n*) were obtained from the reaction of cholesterol-based carboxylic acid with corresponding p-hydroxy benzaldehyde. The tetramesogenic compounds were prepared by heating under reflux 4-(ω-cholesteryloxycarbonylalkonoyloxy) benzaldehyde Cho-*n* (*n* = 2, 4, 6, 8) with 1,4-bis(4-aminophenyl-1-oxy)butane in absolute alcohol with PTSA as catalyst. All the intermediates and final compounds (TSH-*n*) were characterised by FTIR, ^{1}H NMR and the purities of these compounds were verified by thin layer chromatography (TLC). As a representative intermediate, the specific split

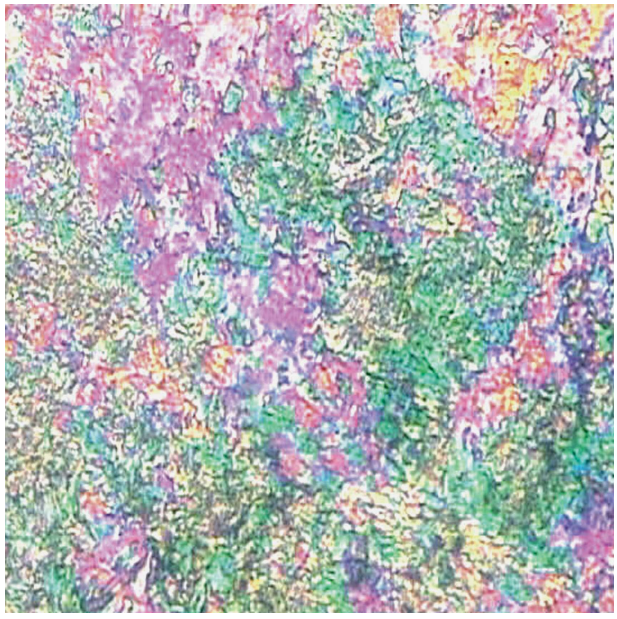
absorptions of compound Cho-2 at 1766, 1736 and 1707 cm^{-1} might be a result of the existence of the three carbonyl groups, namely, two ester C=O and aldehyde C=O stretching bands. Compared with compound Cho-2, the characteristic absorptions of target compound TSH-2 at 1760, 1731 and 1623 cm^{-1} attributed to two ester C=O and imino C=N stretching bands, respectively. The disappearance of the absorption band at 1707 cm^{-1} indicates that the aldehyde group participates in the reaction, while the appearance of an absorption peak at 1623 cm^{-1} might be a result of the formation of the imino group.

3.2 *Liquid crystalline behaviour*

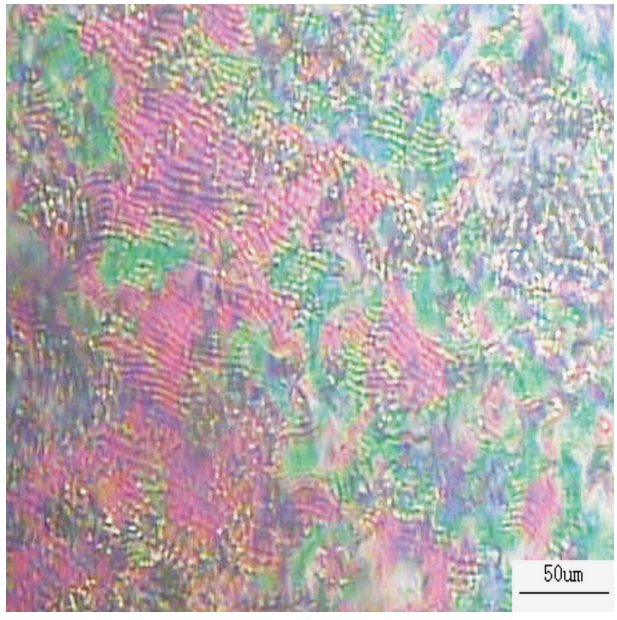
The mesomorphic properties for the intermediate (Cho-*n*) and tetramesogens (TSH-*n*) were investigated by means of a POM equipped with a heating stage and differential scanning calorimeter. The key intermediates Cho-*n* exhibited liquid crystalline properties and displayed the characteristic cholesteric oily-streak texture in the POM observation. The compound Cho-2 had a smectic fan-like texture as well as the expected chiral nematic mesophase with a characteristic oily-streak texture in the heating process. Moreover, they can reflect green colour in the cholesteric state. It is well known that the pitch of chiral nematics is dependent on temperature and selective reflection of light occurs when its wavelength is equal to the pitch of the helical structure in the chiral nematic phase. We did not observe colours other than green on increasing the temperature, probably due to the temperature ranges of the chiral nematic phase being narrow.

The target compound TSH-2 melted at 198°C, and a typical cholesteric Grandjean texture was obtained, as we can see from Figure 1(a). By further heating to 250°C, the LC texture changed to a fingerprint texture with a helical pitch of about 10 μm (Figure 1(b)), and then the birefringence disappeared at 307°C. In addition, the sample began to thermally decompose. Many cholesterol-based compounds in the chiral nematic phase can reflect iridescent colours under the visible light, because the pitch of the helical structure and the wavelength of visible light are of the same order of magnitude, around hundreds of nanometres. However, the compound TSH-2 does not reflect brilliant colours owing to the fact that the pitch of the compound is far greater than the wavelength of visible light.

On heating under the POM, TSH-4, TSH-6 and TSH-8 melted at 185.2, 176.5 and 172°C, respectively, and oily-streak textures appeared. By further heating to 267.5, 246.1 and 225.2°C, they displayed focal-conic fan textures. The samples were converted into the isotropic phase at 292.1, 271 and 250.2°C, respectively, accompanied by decomposition except for compound



(a)

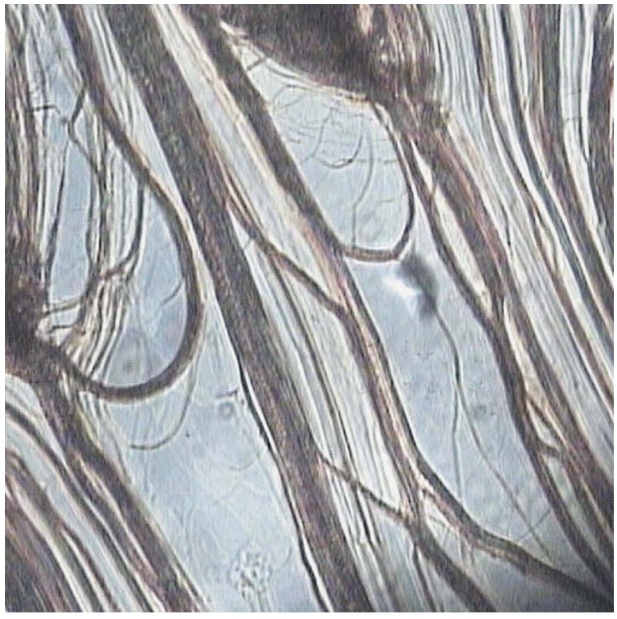


(b)

Figure 1. Optical polarizing micrographs of compound TSH-2 (200×): (a) Grandjean texture at 225°C on heating; (b) fingerprint texture at 250°C on heating.

TSH-8. These textures can be seen in Figure 2(a) and Figure 2(b). The focal-conic fan texture, which on slight mechanical shearing was easily transformed to an oily-streak texture, indicates the presence of the N* phase. We did not perform a cooling run DSC, because of partial decomposition of samples TSH-2, TSH-4 and TSH-6 at their isotropisation temperatures.

The DSC data show slight differences from the observations by POM, because some of the DSC



(a)

(b)

Figure 2. Optical polarizing micrographs of compound TSH-6 (200×): (a) oily-streak texture at 197°C on heating; (b) focal-conic fan texture at 258.9°C on heating.

transition peaks are slightly broad. The DSC traces for TSH-2, TSH-4, TSH-6 and TSH-8 during heating are shown in Figure 3, and display two crystal-to-crystal transitions for TSH-2, TSH-4 and TSH-8, and three crystal-to-crystal transition peaks for compound TSH-6 before mesophase formation, respectively, which could be due to some conformational reorganisation of molecules by passing into a more stable crystalline configuration. Such behaviour has been

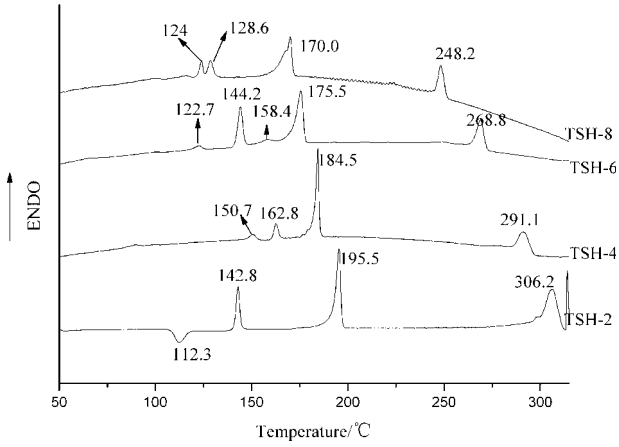


Figure 3. Differential scanning calorimetry traces for the tetramesogenic compounds during heating.

observed in several other mesogens (*36*). One of the latter two peaks in the DSC curves of TSH-2, TSH-4, TSH-6 and TSH-8 represents the melting transition at relatively high temperature, and the other is LC to isotropic (I) phase transition at high temperature.

The phase transition temperatures as well as associated enthalpy changes obtained for the compounds are summarised in Table 1. As can be seen, on increasing the length of the outer alkylene spacer, for tetramers TSH-2 to TSH-8, the melting temperature (T_m) decreases from 195.5°C (TSH-2) to 170°C (TSH-8); at the same time the isotropisation temperature (T_i) reduces from 306.2 to 248.2°C. In addition, the mesogenic phase temperature range (ΔT) also decreases, from 110.7 to 78.25°C, because T_i decreases more quickly than T_m. These observations suggest that the longer the alkylene spacer, the more flexible is the

Table 1. Phase transition temperature (T, °C) and associated transition enthalpy values (ΔH, J g^{-1} in parentheses given for the tetramers TSH-*n* (n = 2, 4, 6, 8).

Compound	Heating	ΔT(°C)
TSH-2	$Cr_1$112.3(−5.56) $Cr_2$142.8(7.64) $Cr_3$195.5(23.79) N*306.2(31.02) I	110.7
TSH-4	$Cr_1$150.7 $Cr_2$162.8 $Cr_3$184.5(27.84)[a] N*291.1(15.97) I	106.6
TSH-6	$Cr_1$122.7 $Cr_2$144.2 $Cr_3$158.4 $Cr_4$175.5(50.83)[a] N*268.8(19.97) I	93.3
TSH-8	$Cr_1$124.0 $Cr_2$128.6(11.94)[b] $Cr_3$170.0(21.75) N*248.2(13.27) I	78.2

Cr, N* and I indicate crystalline state, chiral nematic phase, isotropic liquid, respectively.

[a]Total enthalpy of Cr_1–N* transition.

[b]Total enthalpy of Cr_1–Cr_3 transition, because they cannot be integrated individually. $\Delta T = T_i - T_m$

tetramer. In other words, on increasing the length of spacer, the intermolecular forces are reduced.

In previous studies, liquid crystals containing a cholesteryl unit and a Schiff's base moiety often exhibit rich polymorphic behaviour including SmA, TGB, SmA_{inc}, phases etc., in addition to chiral nematic mesophase (*37*, *38*). In this report, all the tetramesogenic compounds, where the cholesteryl unit and Schiff's base moiety are connected with a polymethylene spacer with two ester bonds, exhibited only a chiral nematic phase. Thus, the formation of liquid crystalline phases in cholesterol-based tetramers may be affected by many factors, such as the chemical nature of linking groups between the spacer and mesogenic units, the length and the parity of spacer, the types of terminal group attached to aromatic rings, the symmetry of liquid crystalline compounds and so on. That there are relatively few studies on liquid crystalline properties for tetramers has prompted us to make a comprehensive and concrete investigation of the chemical structure and physical properties of more chiral tetramers in the future.

4. Conclusions

A series of novel tetramesogenic compounds containing cholesteryl and Schiff base groups were synthesised. All of these tetramesogenic compounds were cholesteric liquid crystals with high phase transition temperatures over a wide range during heating, but they did not exhibit iridescent colours in the liquid crystalline state because the cholesteric pitch length is far greater than the wavelength of visible light. With increasing length of outer alkylene spacers, the melting temperatures and clearing points of the tetramesogenic compounds decreased.

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