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## Liquid Crystals

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## Crystal structure of bis(cholesteryl)4,4'-(1,2-phenylenebis(oxy))-dibutanoate: an oligomesogen

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The crystal structure of bis(cholesteryl)4,4'-(1,2-phenylenebis(oxy))dibutanoate (C<sub>68</sub>H<sub>106</sub>O<sub>6</sub>) has been determined by direct methods using single-crystal X-ray diffraction data. It crystallises in the monoclinic system with space group *P*2<sub>1</sub> and *Z* = 2. The unit cell parameters are *a* = 10.7917(9) Å, *b* = 10.4719(7) Å, *c* = 27.172(2) Å,  $\beta$  = 97.754(3)°. The final reliability factor is *R* = 0.0448 for 4862 observed reflections and the goodness of fit is equal to 1.016. The bond distances and angles are in good agreement with the corresponding values for compounds containing phenyl and cholesterol moieties. The phenyl ring is planar and makes dihedral angles of 52.1(1)° and 85.6(1)° with the cholesterol segments. The cholesterol segments have the usual structure: rings A, C, E, G have chair conformation, and rings B, D, F, H adopt half-chair conformation. The molecules in the unit cell are arranged in layers. The crystal structure is stabilised by inter-molecular C–H...O and C–H... $\pi$  interactions.

**Keywords:** oligomesogen; cholesterol; crystal structure; dihedral angle; C–H... $\pi$  interactions

### 1. Introduction

Of the many varieties of steroids used to generate optically active mesogens and thus chiral mesophases, cholesterol has been used most extensively (1–5). In fact, the first example of a thermotropic mesogen the cholesteryl benzoate (6) is derived from cholesterol. So far, a large number of (over 3000) cholesterol-based systems such as monomers (1–6), oligomers (7–13) and polymers (14) have been reported. The extensive usage of this particular steroid stems from its rigid molecular structure and unique stereochemical configuration having eight chiral centres. Most importantly, the presence of this core in mesogens generally ensures the stabilisation of chiral mesophases, especially the chiral nematic (N\*) phase (1–6). Owing to its periodic helical super-structure the N\* phase has the unique ability to selectively reflect the light in the visible region. In addition, the pitch (*P*) of the helix, which controls the selective reflection wavelength, is extremely sensitive to the external stimuli such as temperature, pressure and electric and magnetic fields (15). As a result, this fluid phase has been used in various technological applications (15, 16). The thin glassy films of the N\* phase, where the helical stacks of birefringent layers are frozen with a fixed pitch length, are of significant interest for a variety of applications, especially for rewritable devices (11–13).

In this context, oligomers comprising cholesterol segment/s have been attracting a great deal of attention given the observation that this steroid aids in quenching the N\* structure upon sudden cooling (11–13). Particularly, oligomesogens consisting of cholesterol moieties joined through a flexible spacer are proven to be promising materials (11–13). These observations

inspired us to design and synthesise several bent-shaped oligomesogens in which two cholesterol entities are joined to an aromatic core through the oxyalkanoyloxy spacer of varying length and parity (17). (The synthesis and thermal behaviour of this compound will be published elsewhere.) Here we report the crystal structure of an oligomesogen formed by covalently linking two cholesterol segments at 1,2-positions of a benzene ring via oxybutanoyloxy spacers. The chemical structure of this compound is shown in Figure 1; the details of synthesis and molecular structural characterisation will be published elsewhere.

The phase behaviour of this oligomesogen was examined with the help of a polarising optical microscope and differential scanning calorimeter. Upon heating it melts into the chiral nematic (N\*) phase at 133°C ( $\Delta H = 55.8 \text{ J g}^{-1}$ ) with the characteristic Grandjean planar texture before transforming into the isotropic liquid phase at 148°C ( $\Delta H = 0.5 \text{ J g}^{-1}$ ). On subsequent cooling, it exhibits a monotropic smectic A (SmA) phase below the N\* phase through a transient twist grain boundary (TGB) phase at about 93°C. The SmA phase showed the focal-conic texture in slides treated for planar orientation and a dark field

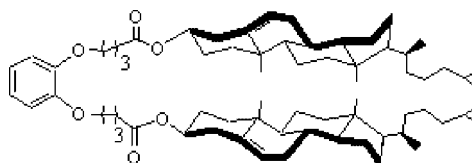


Figure 1. Chemical structure of bis(cholesteryl)4,4'-(1,2-phenylenebis(oxy))dibutanoate.

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of view for homeotropic alignment. On slow heating of the SmA phase shows short-lived filamentary growth in the homeotropic regions, a feature typical of the TGB phase. On further cooling the SmA phase freezes into a glassy state at 40°C instead of crystallisation.

## 2. Experimental details

White transparent rectangular-shaped single crystals of the title compound were grown at room temperature from ethyl acetate by slow evaporation technique. A crystal with dimensions  $0.3 \times 0.2 \times 0.2 \text{ mm}^3$  was chosen for three-dimensional intensity data collection on a Bruker CCD area-detector diffractometer. Data were collected at 100 K. X-ray intensity data of 17,405 reflections (of which 5756 were unique) were recorded and from these 4862 reflections ( $-10 \leq h \leq 10$ ,  $-10 \leq k \leq 10$ ,  $-26 \leq l \leq 26$ ) were treated as observed. The structure was solved by direct methods using SHELXS97 software (18). All non-hydrogen atoms of the molecule were obtained from the E-map. Full-matrix least-squares refinement was carried out using SHELXL97 software (18). Hydrogen atoms were placed at geometrically fixed positions and allowed to ride on the corresponding non-H atoms with C–H = 0.93–0.98 Å, and  $U_{\text{iso}} = 1.5 U_{\text{eq}}$  of the attached C atom for methyl H atoms and  $1.2 U_{\text{eq}}$  for the other H atoms. The final reliability factor is  $R = 0.0448$ , and the goodness of fit is 1.016. The weight was  $1/\sigma^2(F_o^2) + (0.0720P)^2 + 0.3717P$ , where  $P = [F_o^2 + 2F_c^2]/3$ . The minimum and maximum residual electron densities were  $-0.223$  and  $0.275 \text{ e} \text{ \AA}^{-3}$ , respectively. The calculated density is  $1.113 \text{ M g m}^{-3}$ . The linear absorption coefficient is  $\mu = 0.069 \text{ mm}^{-1}$  for MoK $\alpha$  radiation (0.71073 Å). Atomic scattering factors were taken from the International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). Important crystallographic data and refinement parameters are summarised in Table 1. **CCDC-718547** contains the supplementary crystallographic data for this paper.

## 3. Results and discussion

The atomic coordinates and equivalent isotropic displacement parameters are presented in Table 2. Selected bond distances and bond angles are listed in Table 3. An ORTEP view of the molecule is shown in Figure 2 (see also (19)).

The bond distances and angles are in good agreement with the values for compounds containing phenyl and cholesterol moieties (20–23). The average aromatic bond length in the phenyl ring is  $1.369(6) \text{ \AA}$  and the average observed bond angle is  $120.0(4)^\circ$ . The CO double bonds are found to be  $1.189(5) \text{ \AA}$

Table 1. Crystal data and other experimental details.

CCDC number	718547
Crystal description	White transparent rectangular
Crystal size	$0.3 \times 0.2 \times 0.2 \text{ mm}^3$
Empirical formula	$\text{C}_{68}\text{H}_{106}\text{O}_6$
Formula weight	335.49
Radiation (wavelength)	MoK $\alpha$ (0.71073 Å)
Unit cell dimensions	$a = 10.7917(9) \text{ \AA}$ , $b = 10.4719(7) \text{ \AA}$ , $c = 27.172(2) \text{ \AA}$ , $\beta = 97.754(3)^\circ$
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell volume	$3042.7(4) \text{ \AA}^3$
Density (calculated)	$1.113 \text{ M g m}^{-3}$
Number of molecules per unit cell, Z	2
Absorption coefficient	$0.069 \text{ mm}^{-1}$
$F(000)$	1124
Refinement of unit cell	4218 reflections ( $2.08^\circ < \theta < 19.24^\circ$ )
$\theta$ range for entire data collection	$2.27^\circ < \theta < 20.18^\circ$
Reflections collected/unique	17,405/5756
Reflections observed ( $I > 2\sigma(I)$ )	4862
Range of indices	$h = -10$ to $10$ , $k = -10$ to $10$ , $l = -26$ to $26$
Refinement method	Full-matrix least-squares on $F^2$
Number of parameters refined	668
Final R-factor	0.0448
$wR(F^2)$	0.1114
Weight	$[1/\sigma^2(F_o^2) + (0.0720P)^2 + 0.3717P]$ where $P = [F_o^2 + 2F_c^2]/3$
Goodness-of-fit on $F^2$	1.016
$(\Delta/\sigma)_{\text{max}}$	0.002 (for $z \text{ C3}$ )
Final residual electron density	$-0.223 < \Delta\rho < 0.275 \text{ e} \text{ \AA}^{-3}$

(C28 = O2) and  $1.203(5) \text{ \AA}$  (C41=O5), whereas the CO single bonds are  $1.329(5)$  (C28–O1);  $1.439(5) \text{ \AA}$  (O1–C25);  $1.325(5)$  (C41–O6) and  $1.458(5) \text{ \AA}$  (O6–C44). In cholesteryl 4-[4-(4-*n*-butylphenylethynyl)phenoxy]butanoate (DTA-3,4), the respective bonds are found to be  $1.167(8)$ ,  $1.330(7)$  and  $1.459(5) \text{ \AA}$  (see (20)); in cholesteryl 4-[4-(4-*n*-hexylphenylethynyl)phenoxy]butanoate (DTA-3,6), the respective bonds are: molecule A,  $1.163(9)$  (I),  $1.295(8)$ ,  $1.474(8) \text{ \AA}$ ; and molecule B,  $1.207(9)$ ,  $1.297(8)$ ,  $1.447(7) \text{ \AA}$  (see (21)); in cholesteryl 4-[4-(4-*n*-heptylphenylethynyl)phenoxy]butanoate (DTA-3,7), the respective bonds are found to be  $1.186(8)$ ,  $1.308(8)$  and  $1.454(6) \text{ \AA}$  (see (22)).

In the cholesterol moieties of the molecule, the mean bond lengths [C(sp<sup>3</sup>)–C(sp<sup>3</sup>) =  $1.522(6) \text{ \AA}$ ; C(sp<sup>3</sup>)–C(sp<sup>2</sup>) =  $1.498(5) \text{ \AA}$ ] are comparable to the theoretical values as reported by Allen *et al.* (24). The shortening in the C24–C25, C25–C26, C43–C44 and C44–C45 bond lengths may be associated with the substitution at C25 and C44. This feature has also been observed in cholesterol

Table 2. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) for non-hydrogen atoms (estimated standard deviations are given in parentheses).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^*$
O1	0.5261(3)	0.7790(3)	0.05950(11)	0.0609(8)
O2	0.4925(3)	0.5805(3)	0.08345(12)	0.0731(9)
O3	0.0248(3)	0.5531(3)	0.02050(10)	0.0596(8)
O4	-0.1541(2)	0.4102(3)	0.05037(11)	0.0601(8)
O5	0.1734(3)	0.6000(4)	0.16935(12)	0.0885(10)
O6	0.2660(3)	0.4122(3)	0.16265(10)	0.0671(8)
C1	1.9474(5)	1.0526(13)	0.4333(5)	0.181(5)
C2	1.7716(8)	1.1984(9)	0.4628(3)	0.157(4)
C3	1.8119(5)	1.0895(8)	0.4323(4)	0.161(5)
C4	1.7266(4)	1.0269(5)	0.39070(19)	0.0913(16)
C5	1.5923(4)	1.0623(5)	0.38259(17)	0.0754(14)
C6	1.5204(4)	0.9924(5)	0.33990(17)	0.0712(13)
C7	1.3880(4)	1.0386(4)	0.32307(14)	0.0556(11)
C8	1.3095(4)	1.0275(6)	0.36509(16)	0.0790(14)
C9	0.7954(4)	1.0727(4)	0.15710(18)	0.0743(13)
C10	1.1830(4)	1.1492(4)	0.24823(17)	0.0653(13)
C11	1.3301(4)	0.9701(4)	0.27622(14)	0.0494(11)
C12	1.4072(4)	0.9833(4)	0.23311(15)	0.0630(12)
C13	1.3162(4)	0.9804(5)	0.18444(15)	0.0650(12)
C14	1.1940(3)	0.9417(4)	0.20121(14)	0.0472(10)
C15	1.1960(4)	1.0049(4)	0.25245(14)	0.0480(10)
C16	1.0886(4)	0.9459(5)	0.27568(15)	0.0598(12)
C17	0.9653(4)	0.9620(4)	0.24297(14)	0.0596(12)
C18	0.8382(4)	0.9315(4)	0.15720(14)	0.0500(11)
C19	0.9639(3)	0.9113(4)	0.18992(13)	0.0444(10)
C20	1.0753(3)	0.9587(4)	0.16600(13)	0.0450(10)
C21	1.0805(3)	0.8902(4)	0.11766(14)	0.0512(11)
C22	0.9559(4)	0.8735(4)	0.08779(15)	0.0520(11)
C23	0.8477(4)	0.8926(4)	0.10443(14)	0.0463(10)
C24	0.7260(4)	0.8741(4)	0.07047(15)	0.0535(11)
C25	0.6414(4)	0.7877(4)	0.09283(15)	0.0535(11)
C26	0.6166(4)	0.8333(5)	0.14241(16)	0.0632(12)
C27	0.7392(4)	0.8463(5)	0.17669(16)	0.0617(12)
C28	0.4600(4)	0.6718(5)	0.05907(17)	0.0561(11)
C29	0.3427(4)	0.6824(5)	0.02398(18)	0.0691(13)
C30	0.2389(4)	0.5992(4)	0.03814(15)	0.0563(11)
C31	0.1212(4)	0.6294(4)	0.00508(16)	0.0568(11)
C32	-0.0945(4)	0.5777(4)	-0.00056(15)	0.0508(10)
C33	-0.1275(4)	0.6674(5)	-0.03716(17)	0.0647(12)
C34	-0.2490(5)	0.6850(5)	-0.05632(17)	0.0700(13)
C35	-0.3405(5)	0.6164(5)	-0.0393(2)	0.0736(13)
C36	-0.3091(4)	0.5260(5)	-0.00262(18)	0.0673(13)
C37	-0.1870(4)	0.5070(4)	0.01674(15)	0.0535(11)
C38	-0.1741(4)	0.4351(5)	0.10004(18)	0.0675(13)
C39	-0.0652(4)	0.5033(5)	0.12875(17)	0.0689(13)
C40	0.0532(4)	0.4234(5)	0.13408(17)	0.0686(13)
C41	0.1680(5)	0.4895(6)	0.15711(17)	0.0656(13)
C42	0.6120(4)	0.4039(4)	0.19385(15)	0.0640(12)
C43	0.4818(4)	0.3654(4)	0.17216(17)	0.0664(13)
C44	0.3887(4)	0.4636(4)	0.18178(15)	0.0595(12)
C45	0.3978(4)	0.4947(5)	0.23565(15)	0.0641(12)
C46	0.5276(4)	0.5302(5)	0.25757(15)	0.0624(12)
C47	0.5508(5)	0.6411(5)	0.28091(16)	0.0673(13)
C48	0.6760(4)	0.6820(5)	0.30519(17)	0.0664(12)
C49	0.7678(4)	0.5741(4)	0.31266(15)	0.0575(11)
C50	0.7607(4)	0.4946(4)	0.26536(14)	0.0543(11)
C51	0.6286(4)	0.4359(4)	0.24984(14)	0.0578(12)
C52	0.8671(4)	0.3980(5)	0.26710(17)	0.0719(13)

(Continued)

Table 2. (Continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}^*$
C53	0.9951(4)	0.4492(5)	0.28648(17)	0.0710(13)
C54	0.9995(4)	0.5171(4)	0.33646(14)	0.0595(12)
C55	0.8987(4)	0.6211(4)	0.32764(15)	0.0603(12)
C56	0.9243(5)	0.7062(5)	0.37250(18)	0.0813(15)
C57	1.0679(4)	0.7027(5)	0.38599(18)	0.0768(14)
C58	1.1168(4)	0.5953(4)	0.35509(15)	0.0618(12)
C59	0.9716(5)	0.4239(5)	0.37707(17)	0.0800(14)
C60	0.6113(5)	0.3151(5)	0.27995(19)	0.0807(15)
C61	1.2307(4)	0.5296(4)	0.38299(16)	0.0630(12)
C62	1.2764(5)	0.4163(5)	0.35570(19)	0.0901(16)
C63	1.3370(4)	0.6242(5)	0.39616(19)	0.0799(14)
C64	1.4444(5)	0.5766(5)	0.4327(2)	0.0872(15)
C65	1.5419(5)	0.6742(6)	0.4492(2)	0.0898(16)
C66	1.6375(5)	0.6387(6)	0.4924(2)	0.0997(18)
C67	1.7042(6)	0.5172(7)	0.4869(3)	0.145(3)
C68	1.7264(7)	0.7445(7)	0.5082(3)	0.140(3)

$$*U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Table 3. Selected bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) for non-hydrogen atoms (estimated standard deviations are given in parentheses).

O1–C28	1.329(5)	O1–C25	1.439(5)
O2–C28	1.189(5)	O3–C32	1.362(5)
O3–C31	1.419(4)	O4–C37	1.378(5)
O4–C38	1.419(5)	O5–C41	1.203(5)
O6–C41	1.325(5)	O6–C44	1.458(5)
C22–C23	1.323(5)	C24–C25	1.473(5)
C25–C26	1.487(6)	C43–C44	1.486(6)
C44–C45	1.490(6)	C46–C47	1.330(6)
C28–O1–C25	118.4(3)	C32–O3–C31	117.3(3)
C37–O4–C38	115.9(3)	C41–O6–C44	119.2(4)
C20–C14–C13	118.8(3)	C13–C14–C15	104.6(3)
C14–C15–C11	100.1(3)	C53–C54–C58	117.8(4)
C55–C54–C58	102.0(3)	C49–C55–C56	118.5(4)

moieties having substitution at similar position. The bond angles C15–C14–C20, C13–C14–C15, C13–C14–C20, C11–C15–C14, C53–C54–C58, C49–C55–C56, C55–C54–C58 show significant deviations from the ideal tetrahedral value of  $109.4^\circ$  (Table 3). These deviations are common in cholesterol moieties as a result of strain caused by the fusion of five- and six-membered rings, the presence of side chains and bond unsaturations. The bond lengths 1.323(5)  $\text{\AA}$  (C22–C23) and 1.330(6)  $\text{\AA}$  (C46–C47) indicate the double bond nature.

The phenyl ring is perfectly planar (the highest displacement is 0.005(5)  $\text{\AA}$  for the atom C34) and makes dihedral angles of  $52.1(1)^\circ$  and  $85.6(1)^\circ$  with the cholesterol segments. In cholesterol segment I (C11 to C25), ring A has a chair conformation with asymmetry parameters  $\Delta C_S$  (C25) = 2.36,  $\Delta C_2$  (C25–C26) = 2.86 (see (25, 26)). Ring B adopts half-chair conformation with asymmetry parameter  $\Delta C_2$

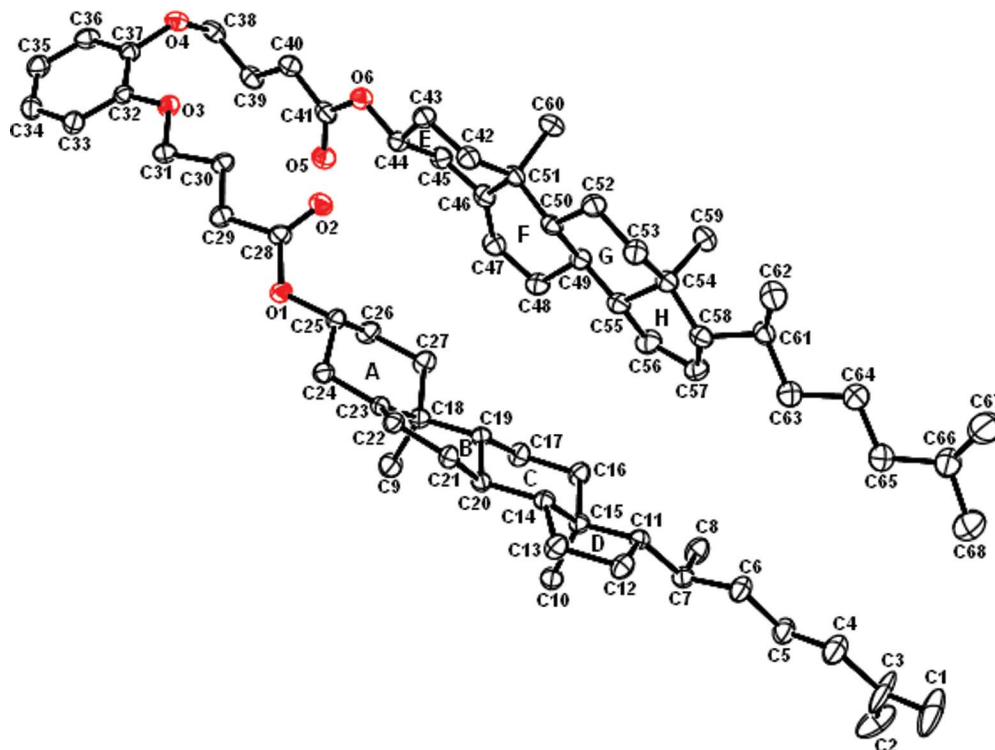


Figure 2. ORTEP view of the molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms have been omitted for clarity.

Table 4. C–H...O and C–H... $\pi$  hydrogen bonding geometry. Cg1 represents the centre of gravity of the phenyl ring.

D–H...A	D–H (Å)	D...A (Å)	H...A (Å)	D–H...A (°)
C21–H21B...O5 <sup>a</sup>	0.97	3.439(6)	2.516(4)	159.0(3)
C24–H24B...Cg1 <sup>a</sup>	0.97	3.903(5)	3.059	146.3
C21–H21A...Cg1 <sup>b</sup>	0.97	3.872(4)	2.939	161.6

Symmetry code: <sup>a</sup>1 + x, y, z; <sup>b</sup>1 – x, 1/2 + y, –z.

(C22–C23) = 2.81. Ring C has a chair conformation with the best rotational axis bisecting C14–C15 and C17–C19 bonds and asymmetry parameter,  $\Delta C_2$  (C14–C15) = 5.1. The best mirror plane passes through C15 and C19, with  $\Delta C_S$  (C15) = 2.05. Ring D is in half-chair conformation ( $\Delta C_2$  (C14–C15) = 6.36). In cholesterol segment II (C44 to C58), ring E has a chair conformation with asymmetry parameters  $\Delta C_S$  (C44) = 0.99,  $\Delta C_2$  (C43–C44) = 2.33. Ring F adopts half-chair conformation with asymmetry parameter  $\Delta C_2$  (C46–C47) = 6.07. Ring G has a chair conformation with the best rotational axis bisecting C50–C52 and C54–C55 bonds and asymmetry parameter,  $\Delta C_2$  (C50–C52) = 4.25. The best mirror plane passes through C50 and C54, with  $\Delta C_S$  (C50) = 4.70.

Ring H is in half-chair conformation ( $\Delta C_2$  (C54–C55) = 3.32).

The pseudo-torsion angles C9–C18...C15–C10 =  $-10.7^\circ$  in cholesterol segment I and C60–C51...C54–C59 =  $-16.0^\circ$  in cholesterol segment II provide a quantitative measure of the twist about the length of the cholesteryl moiety, and show that the cholesteryl moiety in both segments is slightly twisted.

Partial packing of molecules in the unit cell is shown in Figure 3. Projection of the crystal structure along the *a*-axis is shown in Figure 4. From the figure it is clear that the molecules have a distinct smectic-like

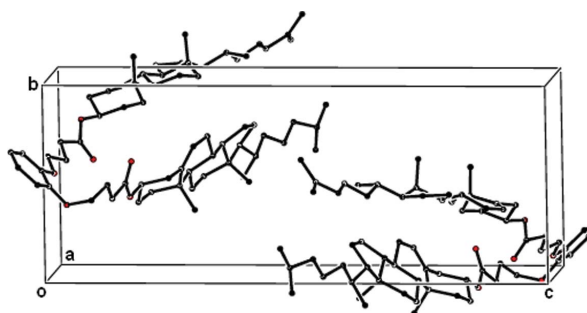


Figure 3. Partial packing of the molecules in the unit cell.

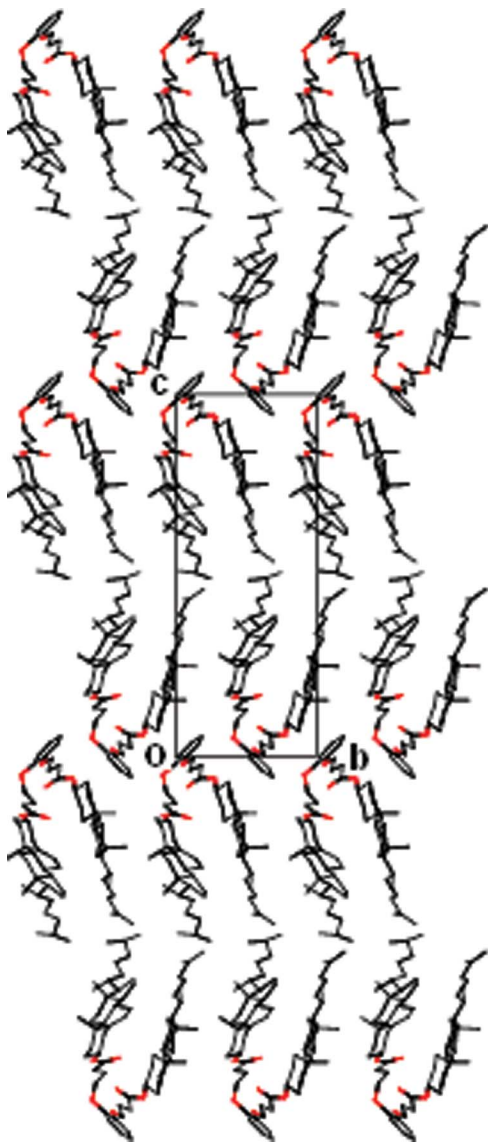


Figure 4. Crystal structure of bis(cholesteryl)4,4'-(1,2-phenylenebis(oxy))dibutanoate projected along the *a*-axis.

structure composed of bilayers. The crystal structure is stabilised by the presence of intermolecular short contacts of the type C–H...O. Two C–H... $\pi$  interactions are also observed which serve to link the molecules in the unit cell (Table 4).

#### Supplementary material

CCDC-718547 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [http://www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

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