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Crystal structure of a liquid crystal non-symmetric dimer: cholesteryl 4-[4-(4-*n*-butylphenylethynyl)phenoxy]butanoate

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The crystal structure of cholesteryl 4-[4-(4-*n*-butylphenylethynyl)phenoxy]butanoate [phase sequence: Cr 155°C (46.1 $\mathrm{J\,g^{-1}}$) SmA 186.8°C (1.5 $\mathrm{J\,g^{-1}}$) TGB-N* 204.7 (6 $\mathrm{J\,g^{-1}}$) I] has been solved from single crystal X-ray diffraction data. The compound crystallizes in the monoclinic space group $P2_1$ with unit cell parameters: a=13.129(2), b=9.3904(10), c=17.4121(8) Å, β=92.790(7)°, Z=2. The structure has been solved by direct methods and refined to R=0.0606 for 3 250 observed reflections. The bond distances and angles are in good agreement with the corresponding values for compounds containing phenyl and cholesterol moieties. The phenyl rings A and B are planar. The dihedral angle between the least-squares planes of the two phenyl rings is 28°. The cholesterol moiety has the usual structure: the C and E rings have chair conformations, and the D and F rings adopt half-chair conformations. The molecules in the unit cell are arranged in an antiparallel manner. The crystal structure is stabilized by an intermolecular C–H…O contact of 2.989(10) Å.

1. Introduction

Cholesterol is a well known naturally occurring steroid, which appears frequently as an important segment in many molecular assemblies, in particular liquid crystals (LCs) [1]. The first reported example of a thermotropic liquid crystal was in fact derived from cholesterol, cholesteryl benzoate [2] and since its discovery many conventional (over 3 000) monomeric LCs containing a cholesteryl ester unit as a chiral part of the molecule have been reported [1 *a*, 3] On the other hand, among non-symmetric oligomesogens, chiral dimers formed by combining a cholesteryl ester entity with various aromatic mesogenic cores through a polymethylene spacer have been attracting attention due to their remarkable thermal behaviour. In particular the dimers comprising a diphenylacetylene segment having an alkoxy tail have shown interesting mesomorphic behaviour. Recently we attached an achiral or a chiral tolane (diphenylacetylene) entity to a cholesteryl ester unit via a central paraffinic spacer, the resulting non-symmetric dimers stabilized the N* [4] or SmA [5] phases over wide temperature ranges. Furthermore we prepared a non-symmetric dimer by joining a cholesteryl ester moiety to a 4-*n*-hexyloxytolane group (diphenylacetylene having an *n*-hexyloxy tail) through an *n*-pentyl (C_5) spacer. Interestingly this dimer showed a reentrant twist grain boundary phase (with smectic A blocks; $\mathrm{TGB_A}$) and a newly discovered twist grain boundary phase (with smectic C* blocks; $\mathrm{TGB_{C*}}$) [6]. More recently Jin *et al.* have synthesized similar compounds but varying in the length of the alkoxy tails, and these are reported to show anomalies of periodicity in different TGB structures [7]. Needless to say that small modifications of the molecular structure in such dimers can drastically influence their thermotropic behaviour. This prompted us to design, synthesize and evaluate the mesomorphic behaviour of new non-symmetric dimers in which an optically active cholesteryl entity is connected to 4-*n*-alkyltolanes instead of 4-*n*-alkoxytolanes. As a consequence we have very recently reported investigations on cholesteryl ω-[4-(4-*n*-alkylphenylethynyl)phenoxy]alkanoates in which the lengths of both the central paraffinic spacer and alkyl tails have been varied [8].

As reported, the lengths of the central alkylene spacer (C_3, C_4, C_5 and C_7) as well as of the alkyl tail (*n*-butyl, *n*-pentyl, *n*-hexyl and *n*-heptyl) have been varied to understand structure–property relationships. The investigations have revealed that all the dimers exhibit smectic A (SmA), twist grain boundary (TGB) and chiral neamtic (N*) phases with the exception of one of

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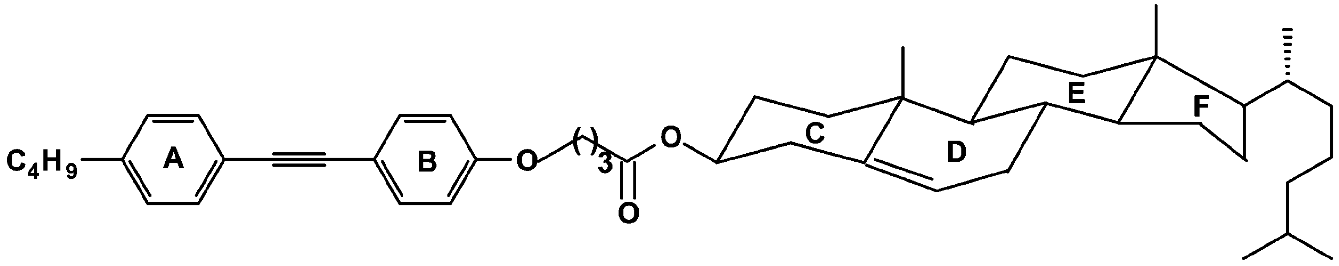

Figure 1. Chemical structure of cholesteryl 4-[4-(4-*n*-butylphenylethynyl)phenoxy]butanoate.

the dimers in which only the N* phase has been stabilized. Some differences in mesomorphic properties of the non-symmetric dimers consisting of odd and even parity alkylene spacers were observed. A point that attracted our attention was the existence of a transient TGB phase. This observation generally casts doubt on the purity of the compound synthesized, as it is possible that the some fine impurities associated with cholesterol would induce TGB phases. However it is our and other investigators' experience that the occurrence of TGB phases is the inherent behaviour of the cholesterol-based non-symmetric dimers. To verify this we have started to elucidate the crystal structures of the cholesteryl ω-[4-(4-*n*-alkylphenylethynyl)phenoxy]alkanoates. We first investigated the first member of the series, namely, cholesteryl 4-[4-(4-*n*-butylphenylethynyl)phenoxy]butanoate (figure 1), which we refer to as DTA-3,4. This compound was synthesized as described earlier [8] and suitable single crystals were grown in a mixture of CH_2Cl_2–EtOH (2/10).

2. Experimental

A crystal having good morphology ($0.3 \times 0.2 \times 0.2\,\text{mm}^3$) was chosen for three-dimensional intensity data collection using an Enraf Nonius CAD-4 diffractometer. CuK_α radiation (λ=1.5418 Å) was used for the data collection. A total of 4 361 reflections were recorded and out of this number 3 250 reflections ($0 \leqslant h \leqslant 15$, $0 \leqslant k \leqslant 11$, $-20 \leqslant l \leqslant 20$) were treated as observed. The structure was solved by direct methods using SHELXS97 [9]. All the non-hydrogen atoms of the molecule were obtained from an E-map. Full-matrix least-squares refinement was carried out using SHELXL97 software [9]. All the hydrogen atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms with C–H=0.96–0.97 Å; $U_{\text{iso}}=1.5\,U_{\text{eq}}$ of the attached C atom for methyl H atoms and $1.2\,U_{\text{eq}}$ for other H atoms. The final refinement cycles converged $R=0.0606$ and $wR(F^2)=0.1950$ [$w=1/\sigma^2(F_o^2)+(0.1409P)^2+0.1717P$] where $P=(F_o^2+2F_c^2)/3$. Maximum shift to e.s.d. ratio for all atoms in the final cycle is 0.005 (for U_{22} C52). Final cycles of refinement resulted in a residual electron density in the range −0.158–0.379 e Å^{-3}. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in table 1. CCDC-247972 contains the supplementary crystallographic data for this paper.

Table 1. Crystal data and other experimental details.

CCDC Number	247972
Crystal description	Colourless plate
Chemical formula	$C_{49}H_{68}O_3$
Molecular mass	705.03
Cell parameters	a=13.129(2) b=9.3904(10) c=17.4121(8) Å β=92.790(7)°
Unit cell volume	2144.1(4) Å^3
Crystal system	Monoclinic
Space group	$P2_1$
Density (calculated)	1.092 Mg m^{-3}
No. of molecules per unit cell, Z	2
Radiation, Wavelength	CuK_α, 1.5418 Å
Absorption coefficient(μ), correction	0.50 mm^{-1}; Psi-scan
Max. and min. transmission	0.9998 and 0.9687
F(000)	772
Refinement of unit cell	25 reflections ($18.2<\theta<23.4°$)
θ range for entire data collection	$2.54<\theta<67.92°$
No. of measured reflections	4361
No. of unique reflections	4163
No. of observed reflections	3250 [I>2 σ (I)]
No. of parameters refined	485
Refinement method	Full- matrix least- squares on F^2
Final R-factor	0.0606
$wR(F^2)$	0.1950
Weight	$1/[\sigma^2(F_o^2)+(0.1409P)^2+0.1717P]$ where $P=[F_o^2+2F_c^2]/3$
Goof (S) on F^2	1.013
Final residual electron density	$-0.158<\Delta\rho<0.379$ e Å^{-3}
$(\Delta/\sigma)_{\max}$ in the final cycle	0.005 (for U_{22} C52)

3. Results and discussion

The final atomic coordinates with equivalent isotropic displacement parameters are listed in table 2. The anisotropic thermal parameters are given in table 3. Selected bond distances, bond angles and torsion angles are listed in table 4. An ORTEP view of the title compound with atomic labelling is shown in figure 2 [10]. The software used to prepare material for publication was SHELXL97 [9].

The bond distances and angles are in good agreement with the values for compounds containing phenyl and cholesterol moieties [11–16]. The average aromatic bond lengths in the phenyl rings A and B are 1.380(8) and 1.377(8) Å, respectively. The average observed bond angle in each of the two phenyl rings is 120.0(5)°. The bond angles C7–C8–C9 and C14–C13–C18 are less than 120°, as was observed also in the related compound, cholesteryl 6[4-(4-pentyloxyphenylethynyl)phenoxy]-hexanoate (DMT5) [13]. The CO double bond (C23–O24) is found to be 1.167(8) Å whereas the CO single bonds are 1.330(7) (C23–O25) and 1.459(5) Å (O25–C26). In 4-cyanobiphenyl-4′-hexylbiphenylcarboxylate, the corresponding bonds are found to be 1.197(5), 1.358(4) and 1.412(4) Å, respectively [17]. The CC triple bond (C11–C12) is found to be 1.201(5) Å and the angles C8–C11–C12 and C13–C11–C12 are 177.2(4)° and 175.8(4)°, respectively. The corresponding values in DMT5 are 1.204(11) Å, 177.4(10)° and 179.3(11)°, respectively.

In the cholesterol moiety of DTA-3,4, the mean bond lengths [$C(sp^3)–C(sp^3)=1.532(6)$ Å; $C(sp^3)–C(sp^2)=1.506(5)$ Å] are comparable to the theoretical values as reported by Allen *et al.* [18]. The bond angles C38–C39–C40, C40–C39–C42, C34–C40–C39, C34–C40–C44, C39–C40–C44, C39–C42–C43 and C39–C42–C45 show significant deviations from the ideal tetrahedral value of 109.4° (table 4). These deviations are common in cholesterol moieties as a result of strain caused by the fusion of five- and six-membered rings, and the presence of side chains and bond unsaturations. The bond length C30–C36 [1.327(5) Å] indicates the double bond nature.

The two phenyl rings A and B are independently planar (highest displacement, −0.011 Å) for the atom C18. However, unlike DMT5, the phenyl rings in DTA-3,4 are not coplanar. The dihedral angle between the two phenyl rings in DMT5 is 4° and in DTA-3,4 it is 28°. The dihedral angles between the planes of the fused rings of the cholesterol moiety and the phenyl rings are 104.7(1)° and 78.5(1)°.

Table 2. Atomic coordinates and equivalent isotropic thermal parameters ($Å^2$) for non-hydrogen atoms (e.s.d. in parenthesis).

Atom	*x*	*y*	*z*	U_{eq}[a]
C1	0.8581(8)	0.3364(15)	0.6566(7)	0.259(9)
C2	0.8121(6)	0.2081(11)	0.6242(7)	0.200(5)
C3	0.7094(5)	0.2347(11)	0.5866(7)	0.180(4)
C4	0.6267(4)	0.1860(10)	0.6277(4)	0.134(2)
C5	0.5210(4)	0.2121(7)	0.5915(3)	0.098(1)
C6	0.4787(5)	0.1169(8)	0.5399(3)	0.116(2)
C7	0.3804(4)	0.1380(7)	0.5058(3)	0.107(2)
C8	0.3252(3)	0.2577(6)	0.5228(2)	0.088(1)
C9	0.3700(3)	0.3544(6)	0.5738(3)	0.094(1)
C10	0.4656(3)	0.3314(6)	0.6075(3)	0.098(1)
C11	0.2228(3)	0.2736(7)	0.4911(2)	0.100(2)
C12	0.1363(3)	0.2810(7)	0.4656(2)	0.101(2)
C13	0.0333(3)	0.2787(7)	0.4353(2)	0.092(2)
C14	−0.0139(4)	0.1486(7)	0.4187(3)	0.101(1)
C15	−0.1141(4)	0.1452(7)	0.3903(3)	0.105(2)
C16	−0.1678(3)	0.2679(6)	0.3775(3)	0.094(1)
C17	−0.1223(4)	0.3965(7)	0.3933(3)	0.101(1)
C18	−0.0226(4)	0.4005(7)	0.4231(3)	0.099(2)
O19	−0.2663(2)	0.2514(5)	0.3481(2)	0.113(1)
C20	−0.3202(4)	0.3752(8)	0.3223(4)	0.128(2)
C21	−0.4204(4)	0.3256(10)	0.2791(4)	0.135(3)
C22	−0.4861(4)	0.2492(9)	0.3294(3)	0.118(2)
C23	−0.5816(4)	0.1962(7)	0.2848(4)	0.111(2)
O24	−0.6061(4)	0.0772(5)	0.2778(4)	0.169(2)
O25	−0.6346(2)	0.3046(4)	0.2542(2)	0.103(1)
C26	−0.7302(3)	0.2744(5)	0.2110(3)	0.085(1)
C27	−0.8174(3)	0.2763(5)	0.2638(2)	0.086(1)
C28	−0.9174(3)	0.2530(5)	0.2175(2)	0.078(1)
C29	−0.9377(3)	0.3619(4)	0.1510(2)	0.066(1)
C30	−0.8432(3)	0.3722(4)	0.1049(2)	0.070(1)
C31	−0.7423(3)	0.3879(5)	0.1496(3)	0.085(1)
C32	−0.9592(3)	0.5098(5)	0.1865(3)	0.085(1)
C33	−1.0297(2)	0.3093(4)	0.0998(2)	0.063(1)
C34	−1.0382(3)	0.3827(4)	0.0209(2)	0.063(1)
C35	−0.9413(3)	0.3590(5)	−0.0222(2)	0.072(1)
C36	−0.8464(3)	0.3699(4)	0.0286(2)	0.074(1)
C37	−1.1319(3)	0.3130(5)	0.1395(2)	0.080(1)
C38	−1.2229(3)	0.2613(5)	0.0892(2)	0.078(1)
C39	−1.2335(2)	0.3437(4)	0.0134(2)	0.064(1)
C40	−1.1310(2)	0.3272(3)	−0.0249(2)	0.061(1)
C41	−1.2597(3)	0.5005(5)	0.0280(3)	0.084(1)
C42	−1.3070(3)	0.2813(4)	−0.0501(2)	0.068(1)
C43	−1.2639(3)	0.3358(5)	−0.1255(2)	0.079(1)
C44	−1.1532(3)	0.3829(5)	−0.1068(2)	0.075(1)
C45	−1.4228(3)	0.3099(5)	−0.0460(2)	0.081(1)
C46	−1.4654(3)	0.2603(10)	0.0283(3)	0.125(2)
C47	−1.4830(3)	0.2452(6)	−0.1151(3)	0.089(1)
C48	−1.5889(3)	0.3051(6)	−0.1278(3)	0.097(1)
C49	−1.6508(3)	0.2427(5)	−0.1955(2)	0.084(1)
C50	−1.7585(4)	0.2974(7)	−0.2041(4)	0.117(2)
C51	−1.8179(5)	0.2213(9)	−0.2701(5)	0.141(3)
C52	−1.7670(8)	0.4505(9)	−0.2132(5)	0.212(6)

[a] $U_{eq}=(1/3)\ \Sigma_i\Sigma_j U_{ij}\ a_i^* a_j^* \mathbf{a_i}.\mathbf{a_j}$.

Ring C has an ideal chair conformation with the best rotational axis bisecting C26–C27 and C29–C30 bonds and with the asymmetry parameter $\Delta C_2(C26–C27)=1.79$ [19, 20]. The best mirror plane passes through C26 and C29, with $\Delta Cs(C26)=3.5$. Ring D adopts a half-chair conformation with the rotational axis bisecting the

Table 3. Anisotropic thermal parameters (Å^2) for non-hydrogen atoms (e.s.d. in parenthesis).

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.181(10)	0.310(2)	0.279(14)	0.097(15)	−0.089(10)	−0.090(12)
C2	0.095(4)	0.172(8)	0.327(14)	0.003(9)	−0.069(7)	0.036(5)
C3	0.083(4)	0.170(7)	0.282(10)	0.081(8)	−0.038(5)	−0.004(4)
C4	0.099(4)	0.175(7)	0.129(4)	0.042(5)	−0.004(3)	0.022(4)
C5	0.076(3)	0.119(4)	0.099(3)	0.022(3)	−0.001(2)	0.001(3)
C6	0.108(4)	0.117(4)	0.125(4)	−0.006(4)	0.015(3)	0.017(3)
C7	0.100(3)	0.118(4)	0.102(3)	−0.021(3)	−0.002(3)	−0.006(3)
C8	0.072(2)	0.118(4)	0.073(2)	0.002(2)	0.002(2)	−0.014(2)
C9	0.068(2)	0.111(4)	0.101(3)	−0.008(3)	−0.004(2)	−0.007(2)
C10	0.078(2)	0.113(4)	0.100(3)	−0.008(3)	−0.011(2)	−0.010(3)
C11	0.079(3)	0.139(4)	0.081(2)	0.007(3)	−0.003(2)	−0.018(3)
C12	0.077(2)	0.144(4)	0.080(2)	−0.002(3)	−0.009(2)	−0.017(3)
C13	0.072(2)	0.128(4)	0.074(2)	−0.005(3)	−0.008(2)	−0.019(3)
C14	0.084(3)	0.110(4)	0.109(3)	−0.009(3)	−0.010(3)	−0.004(3)
C15	0.074(3)	0.111(4)	0.129(4)	−0.010(3)	−0.010(3)	−0.014(3)
C16	0.067(2)	0.108(3)	0.104(3)	−0.009(3)	−0.011(2)	−0.018(3)
C17	0.086(3)	0.111(4)	0.104(3)	0.003(3)	−0.020(3)	−0.015(3)
C18	0.081(3)	0.113(4)	0.102(3)	−0.002(3)	−0.017(2)	−0.028(3)
O19	0.070(2)	0.121(3)	0.146(3)	−0.007(2)	−0.023(2)	−0.016(2)
C20	0.085(3)	0.127(5)	0.167(5)	0.031(4)	−0.032(3)	−0.017(3)
C21	0.092(3)	0.182(7)	0.130(4)	0.046(5)	−0.021(3)	−0.010(4)
C22	0.083(3)	0.150(5)	0.118(3)	0.021(4)	−0.017(3)	−0.004(3)
C23	0.080(3)	0.095(4)	0.153(5)	0.015(3)	−0.037(3)	−0.004(3)
O24	0.135(4)	0.101(3)	0.260(6)	0.021(3)	−0.104(4)	−0.002(3)
O25	0.081(2)	0.083(2)	0.140(2)	−0.004(2)	−0.042(2)	−0.004(2)
C26	0.072(2)	0.076(2)	0.103(3)	−0.009(2)	−0.027(2)	0.004(2)
C27	0.084(2)	0.086(3)	0.086(2)	−0.004(2)	−0.016(2)	0.005(2)
C28	0.073(2)	0.079(2)	0.081(2)	−0.002(2)	−0.008(2)	0.000(2)
C29	0.063(2)	0.061(2)	0.073(2)	−0.007(2)	−0.001(1)	0.002(2)
C30	0.060(2)	0.061(2)	0.089(2)	−0.000(2)	−0.002(2)	0.002(2)
C31	0.066(2)	0.084(3)	0.104(3)	−0.008(2)	−0.009(2)	−0.001(2)
C32	0.078(2)	0.071(2)	0.105(3)	−0.019(2)	−0.014(2)	0.008(2)
C33	0.061(2)	0.060(2)	0.069(2)	−0.004(1)	0.001(1)	−0.002(1)
C34	0.059(2)	0.056(2)	0.073(2)	0.002(1)	0.004(1)	−0.001(1)
C35	0.062(2)	0.080(2)	0.074(2)	0.004(2)	0.010(2)	−0.001(2)
C36	0.056(2)	0.074(2)	0.092(2)	0.001(2)	0.007(2)	−0.004(2)
C37	0.066(2)	0.106(3)	0.067(2)	0.000(2)	0.005(2)	−0.010(2)
C38	0.066(2)	0.099(3)	0.070(2)	0.003(2)	0.008(2)	−0.016(2)
C39	0.055(2)	0.066(2)	0.073(2)	−0.002(2)	0.002(1)	−0.004(1)
C40	0.060(2)	0.056(2)	0.066(2)	0.002(1)	0.002(1)	0.000(1)
C41	0.066(2)	0.077(2)	0.108(3)	−0.023(2)	−0.002(2)	0.008(2)
C42	0.065(2)	0.064(2)	0.074(2)	−0.002(2)	0.001(1)	−0.002(2)
C43	0.073(2)	0.082(2)	0.080(2)	0.007(2)	−0.002(2)	−0.001(2)
C44	0.065(2)	0.085(2)	0.076(2)	0.016(2)	0.001(2)	−0.000(2)
C45	0.061(2)	0.091(3)	0.091(2)	−0.014(2)	−0.001(2)	−0.008(2)
C46	0.066(2)	0.205(7)	0.105(3)	−0.009(4)	0.006(2)	−0.031(4)
C47	0.066(2)	0.102(3)	0.098(3)	−0.008(2)	−0.003(2)	−0.011(2)
C48	0.074(2)	0.096(3)	0.120(3)	−0.023(3)	−0.014(2)	−0.001(2)
C49	0.067(2)	0.086(3)	0.097(2)	0.003(2)	−0.005(2)	−0.009(2)
C50	0.093(3)	0.127(5)	0.128(4)	−0.012(4)	−0.017(3)	0.007(3)
C51	0.088(4)	0.135(6)	0.195(7)	0.002(5)	−0.043(4)	−0.022(4)
C52	0.239(11)	0.193(10)	0.190(8)	−0.090(7)	−0.118(8)	0.116(9)

Table 4. Selected bond distances (Å) bond angles (°) and torsion angles (°) for non-hydrogen atoms (e.s.d. in parentheses).

C11–C12	1.202(6)	O25–C26	1.459(4)
C22–O24	1.167(7)	C30–C36	1.326(6)
C23–O25	1.330(6)		
C7–C8–C9	117.6(4)	C34–C40–C44	118.9(3)
C12–C11–C8	177.2(7)	C34–C40–C39	115.7(3)
C11–C12–C13	175.8(7)	C44–C40–C39	103.8(3)
C18–C13–C14	118.0(4)	C43–C42–C39	103.9(3)
C38–C39–C40	106.2(3)	C39–C42–C45	118.8(3)
C40–C39–C42	100.4(3)		
C31–C26–C27–C28	58.7(5)	C34–C35–C36–C30	12.6(6)
C26–C27–C28–C29	−56.1(5)	C34–C33–C37–C38	50.4(5)
C27–C28–C29–C30	48.8(4)	C33–C37–C38–C39	−55.3(5)
C33–C29–C30–C36	15.0(5)	C37–C38–C39–C40	56.1(4)
C28–C29–C30–C31	−46.7(4)	C33–C34–C40–C39	56.9(4)
C27–C26–C31–C30	−55.9(5)	C38–C39–C40–C34	−59.6(4)
C29–C30–C31–C26	51.4(5)	C42–C39–C40–C44	46.1(3)
C30–C29–C33–C34	−44.3(4)	C40–C39–C42–C43	−38.7(3)
C37–C33–C34–C40	−49.0(4)	C39–C42–C43–C44	17.9(4)
C29–C33–C34–C35	58.2(4)	C39–C40–C44–C43	−35.2(4)
C33–C34–C35–C36	−40.5(4)	C42–C43–C44–C40	10.4(4)
C29–C30–C36–C35	0.8(6)		

C30–C36 and C33–C34 bonds and with the asymmetry parameter ΔC_2 (C30–C36)=4.84. Ring E is in a normal chair conformation with atoms C33 and C39 situated 0.616(4) and 0.717(4) Å, respectively, above and below the plane defined by the other four ring atoms. The asymmetry parameters are: ΔC_2 (C33–C37)=4.5; ΔC_s(C33)=2.38. The conformation of ring F is 39α–40β half-chair [ΔC_2 (C39–C40)=5.88] with a phase angle of pseudorotation $\Delta=8.95°$ and maximum angle of torsion $\Phi_m=46.24°$ [21].

The packing of DTA-3,4 molecules in the unit cell is shown in figure 3. From this figure it is evident that the molecules in the unit cell are arranged in an antiparallel manner. The molecule is extended; this may be attributed to the presence of a triple bond between the phenyl groups which reduces the flexibility. Projections

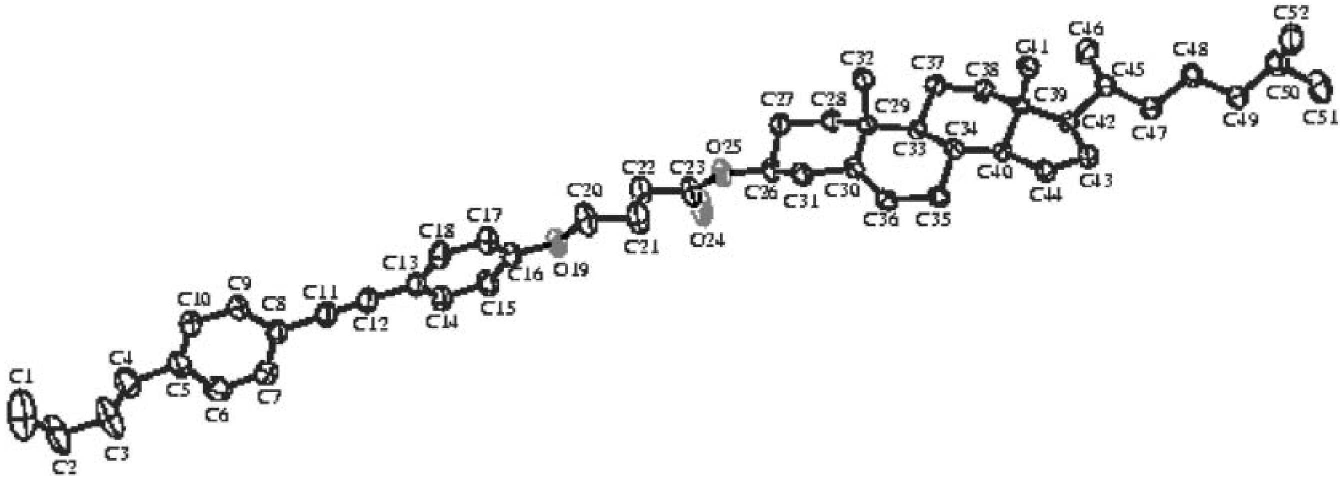

Figure 2. ORTEP view of the DTA-3,4 molecule with displacement ellipsoids drawn at 20% probability level.

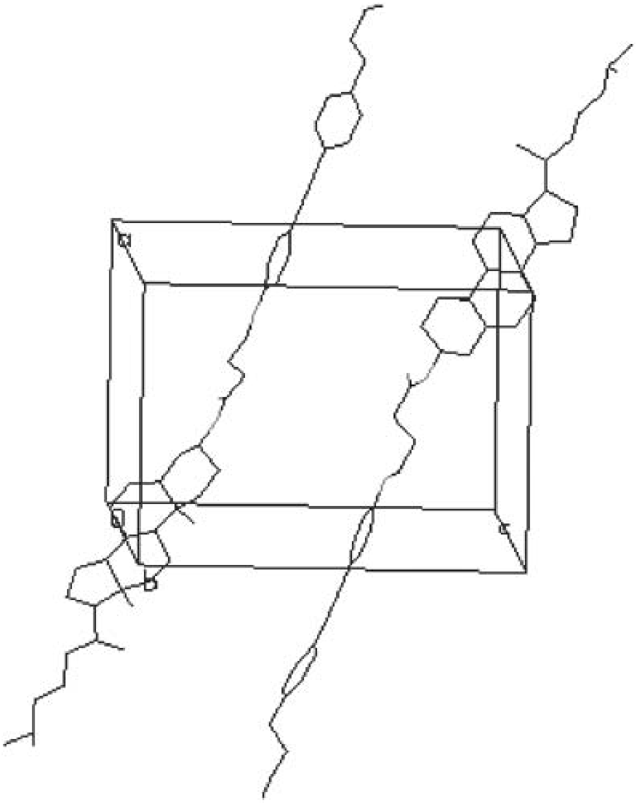

Figure 3. Partial packing of DTA-3,4 molecules in the unit cell.

of the crystal structure along the *a*, *b* and *c* axes are shown, respectively, in figures 4, 5 and 6. From these figures it is evident that the molecules related by the two-fold screw axis are packed in interpenetrating layers. The crystal structure is stabilized by the presence of intermolecular short contact of the type C–H…O involving O24 of the ester group, viz. C4…O24 2.989(10), H4B…O24 2.183(6) Å, C4-H4B…O24 139.6(5)° (Symmetry: $-x,-y, -z+1$). There also exist two intramolecular short contacts with C22–H22B…O19 = 2.888(6) and C26–H26…O24 = 2.693(7) Å, which provide molecular stability in the unit cell.

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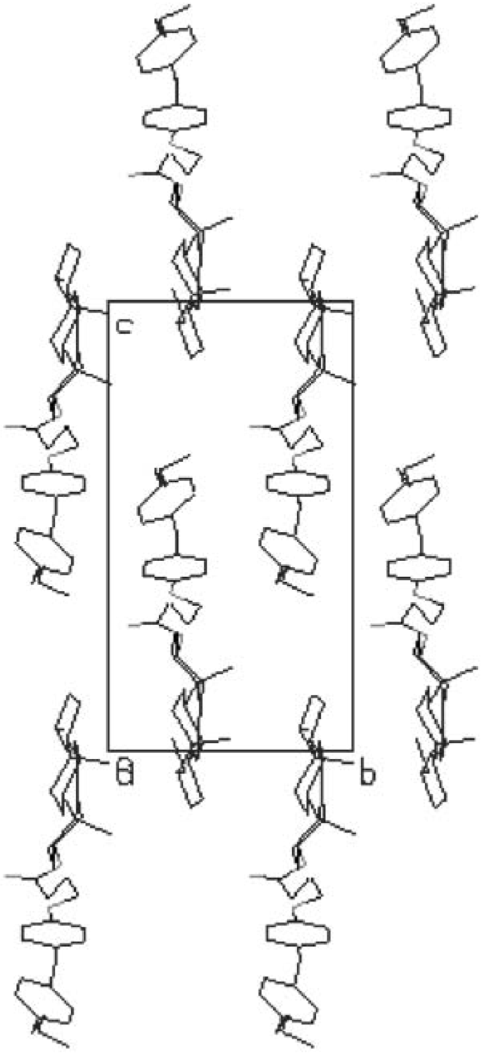

Figure 4. Crystal structure of DTA-3,4 projected along the *a*-axis.

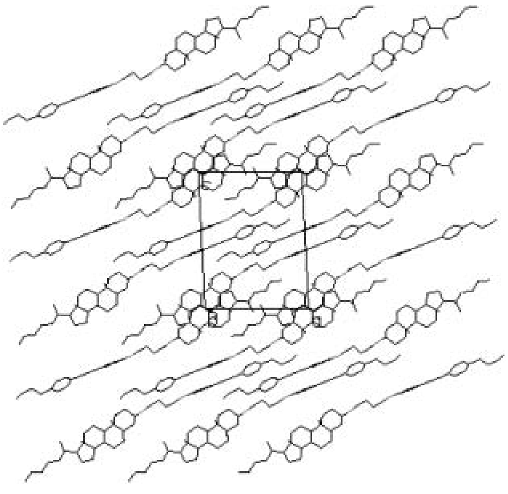

Figure 5. Crystal structure of DTA-3,4 projected along the *b*-axis.

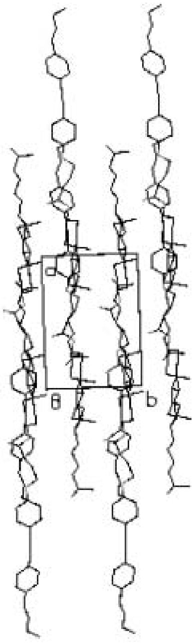

Figure 6. Crystal structure of DTA-3,4 projected along the *c*-axis.

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