C6	0.4736 (2)	0.1077 (3)	1.14838 (14)	0.0639 (6)
C7	0.4273 (2)	−0.0328 (3)	1.08669 (13)	0.0546 (5)
O7	0.46299 (13)	−0.1800 (2)	1.13346 (11)	0.0705 (4)
C8	0.3493 (2)	−0.0178 (2)	0.98327 (13)	0.0527 (5)
C10	0.3638 (2)	0.2798 (2)	0.99995 (15)	0.0572 (5)
C11	−0.2110 (3)	0.1647 (4)	0.3367 (2)	0.1029 (10)
C21	0.1525 (2)	0.3363 (3)	0.6008 (2)	0.0841 (8)
C22	0.0748 (2)	0.3225 (4)	0.4986 (2)	0.0795 (7)
C23	−0.0312 (2)	0.2513 (3)	0.4725 (2)	0.0666 (6)
O23	−0.1017 (2)	0.2397 (2)	0.36774 (12)	0.0870 (5)
C24	−0.0602 (2)	0.2005 (4)	0.5492 (2)	0.0926 (9)
C25	0.0184 (3)	0.2177 (4)	0.6522 (2)	0.1135 (11)

† Site occupancy = 0.506 (5).
‡ Site occupancy = 0.494 (5).

Table 2. *Selected geometric parameters* (Å, °)

O1—C9	1.360 (2)	C20—C25	1.375 (4)
O1—C2	1.400 (5)	C5—C6	1.358 (3)
C2—C20	1.515 (5)	C5—C10	1.390 (3)
C2′—C20	1.595 (5)	C6—C7	1.403 (3)
C2—C3	1.528 (7)	C7—O7	1.346 (3)
C2′—C3′	1.498 (8)	C7—C8	1.376 (2)
C3—C4	1.576 (5)	C11—O23	1.412 (3)
C3′—C4	1.476 (7)	C21—C22	1.361 (3)
C4—O4	1.225 (3)	C22—C23	1.372 (3)
C4—C10	1.440 (3)	C23—C24	1.361 (4)
C9—C8	1.385 (3)	C23—O23	1.367 (3)
C9—C10	1.399 (3)	C24—C25	1.376 (3)
C20—C21	1.374 (4)		
C9—O1—C2	118.3 (2)	O1—C2′—C20	101.1 (3)
C9—O1—C2′	113.79 (3)	O1—C2′—C3′	113.5 (4)
O1—C2—C20	108.2 (3)	C4—C3′—C2′	104.7 (4)
O1—C2—C3	105.8 (4)	C6—C5—C10	122.2 (2)
C20—C2—C3	104.8 (4)	C5—C6—C7	119.1 (2)
C2—C3—C4	108.6 (4)	O7—C7—C8	122.6 (2)
O4—C4—C10	123.8 (2)	O7—C7—C6	116.9 (2)
O4—C4—C3	122.4 (3)	C8—C7—C6	120.6 (2)
C10—C4—C3	112.1 (3)	C7—C8—C9	119.3 (2)
C3′—C4—C10	116.1 (3)	C5—C10—C9	117.7 (2)
C3′—C4—O4	117.9 (3)	C5—C10—C4	122.6 (2)
O1—C9—C8	116.5 (2)	C9—C10—C4	119.7 (2)
O1—C9—C10	122.4 (2)	C22—C21—C20	121.5 (3)
C8—C9—C10	121.2 (2)	C21—C22—C23	120.1 (3)
C21—C20—C25	117.5 (2)	C24—C23—O23	124.7 (2)
C21—C20—C2	108.1 (3)	C24—C23—C22	119.8 (2)
C2′—C20—C21	132.9 (3)	O23—C23—C22	115.5 (2)
C2′—C20—C25	108.7 (3)	C23—O23—C11	117.7 (2)
C25—C20—C2	133.5 (3)	C23—C24—C25	119.4 (3)
C20—C2′—C3′	112.8 (4)	C20—C25—C24	121.7 (3)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: AB1220). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cholesteryl 4-(8-Octenyloxy)benzoate

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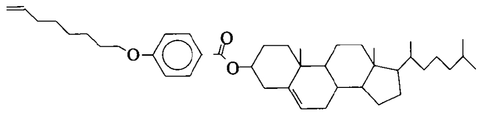
Abstract

The crystal structure of cholest-5-en-3-yl 4-(8-octenyloxy)benzoate, $C_{42}H_{64}O_3$, was determined by single-crystal X-ray diffraction. The benzoate moiety is twisted 64.6 (2)° from the least-squares plane through the tetracyclic core. Elongated molecules pack in an antiparallel fashion. Fully overlapping molecules are related by 2_1 screw symmetry.

Comment

The title structure, (I), is the second in a series of crystallographic studies of cholesteryl *n*-alkenebenzoate compounds (Socci, Farmer, Chabinyc, Fratini, Bunning & Adams, 1995). When these compounds are attached to cyclosiloxane backbones, a class of intermediate molecular weight liquid crystals is produced (Bunning, 1992; Bunning, Klei, Samulski, Crane & Adams, 1993). Ordered transparent films of these materials have potential use as matrices for optical devices. An understanding of the solid-state structure of cholesteryl esters may provide an insight into the types of packing exhibited by these molecules in the liquid-crystalline state. Relationships between the crystal structures and the packing in the liquid-crystalline states of cholesteryl esters have been observed (Sawzik & Craven, 1979). In the present work, we observe similarities between the packing in the solid and liquid-crystalline states.



(I)

A displacement ellipsoid plot of the title molecule (without H atoms), along with the numbering scheme employed, is shown in Fig. 1. The conformation of the tetracyclic core is consistent with other cholesterol derivatives (Guerina & Craven, 1979; Sawzik & Craven, 1979). The benzoate moiety is twisted out of the least-squares plane through the tetracyclic core by 64.6 (2)°. The octenyloxy chain is nearly extended and slightly bowed from the plane of the benzoate group. The terminal vinyl torsion angle, C39—C40—C41—C42, is −105 (1)°. The intermolecular distances about atom C42 are normal. The largest r.m.s. amplitude thermal vibrations are observed in the terminal methyl atoms, C26 and C27, of the aliphatic tail and in the terminal vinyl group, C41 and C42, of the octenyloxy chain. This behavior has been observed in other cholesteryl esters (Sawzik & Craven, 1980; Sato & Yano, 1987; Socci *et al.*, 1995).

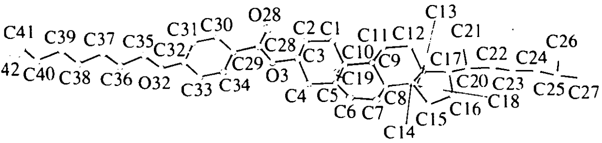


Fig. 1. The molecular structure of the title compound (*ORTEP*II; Johnson, 1976) along with the atom-numbering scheme. H atoms are omitted and displacement ellipsoids are drawn at the 50% probability level.

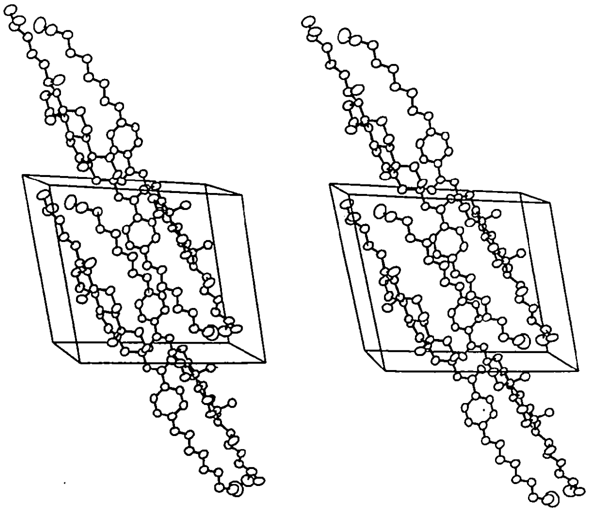


Fig. 2. A stereo *ORTEP*II (Johnson, 1976) plot of the unit cell. The *b* axis is nearly perpendicular to the plane of the paper and the *c* and *a* axes are horizontal and vertical, respectively.

The molecules are packed antiparallel, as shown in the depiction of the unit cell (Fig. 2). The long axis of the molecule makes an angle of 38° with the (100) plane. The phenyl ring and the tetracyclic core of fully overlapping molecules are approximately orthogonal. The end-to-end spacing between these two molecules corresponds well to the layer spacings observed in the smectic liquid-crystalline phase.

Experimental

The synthesis of the title compound is described in Gresham *et al.* (1994).

Crystal data

$C_{42}H_{64}O_3$
$M_r = 616.93$
Monoclinic
$P2_1$
$a = 15.134\,(2)$ Å
$b = 7.403\,(2)$ Å
$c = 17.270\,(2)$ Å
$\beta = 105.25\,(1)°$
$V = 1866.7\,(6)$ Å^3
$Z = 2$
$D_x = 1.098$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.33–13.88°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Parallelepiped
0.45 × 0.36 × 0.30 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration from crystal shape
$T_{min} = 0.9758$, $T_{max} = 0.9855$
8455 measured reflections
5822 independent reflections
2545 observed reflections [$I > 2.0\sigma(I)$]

$R_{int} = 0.058$
$\theta_{max} = 29.97°$
$h = -21 \rightarrow 20$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 24$
3 standard reflections monitored every 120 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.059$
$wR = 0.050$
$S = 1.549$
2545 reflections
597 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + 0.00025F^2]$

$(\Delta/\sigma)_{max} = 0.1$
$\Delta\rho_{max} = 0.721$ e Å^{-3}
$\Delta\rho_{min} = -0.516$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O3	0.0612 (2)	0.2589 (8)	0.6159 (2)	0.052 (2)
O28	0.0086 (2)	0.303 (1)	0.4834 (2)	0.077 (2)
O32	0.4403 (2)	0.3283 (9)	0.5345 (2)	0.061 (2)

C1	−0.1693 (4)	0.383 (1)	0.6460 (4)	0.058 (3)
C2	−0.0775 (4)	0.412 (1)	0.6266 (4)	0.063 (4)
C3	−0.0313 (3)	0.234 (1)	0.6233 (3)	0.050 (3)
C4	−0.0209 (3)	0.126 (1)	0.7001 (3)	0.054 (3)
C5	−0.1107 (3)	0.108 (1)	0.7235 (3)	0.046 (3)
C6	−0.1377 (4)	−0.051 (1)	0.7423 (3)	0.050 (3)
C7	−0.2222 (4)	−0.082 (1)	0.7701 (3)	0.050 (3)
C8	−0.2632 (3)	0.092 (1)	0.7931 (3)	0.044 (3)
C9	−0.2608 (3)	0.2414 (9)	0.7324 (3)	0.039 (2)
C10	−0.1627 (3)	0.283 (1)	0.7249 (3)	0.044 (3)
C11	−0.3120 (4)	0.411 (1)	0.7473 (4)	0.062 (3)
C12	−0.4092 (4)	0.374 (1)	0.7596 (4)	0.054 (3)
C13	−0.4043 (3)	0.232 (1)	0.8263 (3)	0.045 (3)
C14	−0.3597 (3)	0.065 (1)	0.7998 (3)	0.043 (3)
C15	−0.3784 (4)	−0.083 (1)	0.8543 (4)	0.058 (3)
C16	−0.4728 (4)	−0.038 (1)	0.8661 (4)	0.059 (3)
C17	−0.4966 (3)	0.154 (1)	0.8321 (3)	0.046 (3)
C18	−0.3491 (4)	0.304 (1)	0.9070 (3)	0.065 (3)
C19	−0.1089 (4)	0.399 (1)	0.7968 (4)	0.066 (4)
C20	−0.5537 (3)	0.260 (1)	0.8794 (3)	0.052 (3)
C21	−0.5721 (5)	0.455 (1)	0.8525 (5)	0.079 (4)
C22	−0.6460 (4)	0.164 (1)	0.8721 (4)	0.057 (3)
C23	−0.6970 (4)	0.223 (1)	0.9315 (4)	0.062 (3)
C24	−0.7856 (4)	0.124 (1)	0.9241 (4)	0.067 (4)
C25	−0.8363 (4)	0.172 (1)	0.9865 (4)	0.085 (5)
C26	−0.8664 (5)	0.367 (2)	0.9846 (5)	0.102 (6)
C27	−0.9183 (6)	0.046 (2)	0.9779 (6)	0.122 (7)
C28	0.0723 (3)	0.287 (1)	0.5424 (3)	0.053 (3)
C29	0.1695 (3)	0.305 (1)	0.5417 (3)	0.045 (3)
C30	0.2418 (3)	0.296 (1)	0.6109 (3)	0.050 (3)
C31	0.3304 (3)	0.306 (1)	0.6066 (3)	0.049 (3)
C32	0.3503 (3)	0.322 (1)	0.5334 (3)	0.052 (3)
C33	0.2790 (3)	0.333 (1)	0.4641 (3)	0.053 (3)
C34	0.1898 (3)	0.324 (1)	0.4695 (3)	0.050 (3)
C35	0.4648 (3)	0.330 (1)	0.4597 (3)	0.057 (3)
C36	0.5658 (4)	0.326 (1)	0.4739 (3)	0.058 (3)
C37	0.5942 (4)	0.318 (1)	0.3969 (3)	0.059 (3)
C38	0.6957 (3)	0.307 (1)	0.4059 (3)	0.054 (3)
C39	0.7192 (3)	0.296 (1)	0.3263 (3)	0.061 (3)
C40	0.8188 (4)	0.274 (1)	0.3297 (4)	0.072 (4)
C41	0.8379 (5)	0.261 (2)	0.2517 (5)	0.103 (6)
C42	0.8585 (8)	0.138 (2)	0.2127 (7)	0.160 (9)

Table 2. *Selected geometric parameters* (Å, °)

O3—C3	1.450 (6)	C15—C16	1.53 (1)
O3—C28	1.340 (6)	C16—C17	1.54 (1)
O28—C28	1.210 (5)	C17—C20	1.548 (9)
O32—C32	1.359 (6)	C20—C21	1.52 (1)
O32—C35	1.435 (7)	C20—C22	1.545 (8)
C1—C2	1.527 (9)	C22—C23	1.50 (1)
C1—C10	1.530 (9)	C23—C24	1.51 (1)
C2—C3	1.50 (1)	C24—C25	1.52 (1)
C3—C4	1.523 (9)	C25—C26	1.51 (2)
C4—C5	1.522 (8)	C25—C27	1.53 (1)
C5—C6	1.32 (1)	C28—C29	1.480 (7)
C5—C10	1.519 (9)	C29—C30	1.393 (6)
C6—C7	1.497 (9)	C29—C34	1.368 (7)
C7—C8	1.53 (1)	C30—C31	1.365 (7)
C8—C9	1.531 (9)	C31—C32	1.379 (8)
C8—C14	1.509 (8)	C32—C33	1.386 (6)
C9—C10	1.554 (7)	C33—C34	1.379 (7)
C9—C11	1.53 (1)	C35—C36	1.483 (8)
C10—C19	1.554 (9)	C36—C37	1.502 (9)
C11—C12	1.564 (9)	C37—C38	1.506 (8)
C12—C13	1.547 (9)	C38—C39	1.511 (8)
C13—C14	1.533 (9)	C39—C40	1.501 (8)
C13—C17	1.539 (8)	C40—C41	1.46 (1)
C13—C18	1.520 (8)	C41—C42	1.22 (2)
C14—C15	1.52 (1)		
C3—O3—C28	117.9 (3)	C13—C14—C15	103.2 (5)
C32—O32—C35	119.0 (3)	C14—C15—C16	104.9 (6)
C2—C1—C10	114.4 (4)	C15—C16—C17	106.5 (6)
C1—C2—C3	110.3 (6)	C13—C17—C16	104.1 (5)
O3—C3—C2	111.4 (6)	C13—C17—C20	119.2 (6)
O3—C3—C4	105.2 (4)	C16—C17—C20	111.8 (5)
C2—C3—C4	111.8 (5)	C17—C20—C21	113.7 (6)
C3—C4—C5	112.4 (4)	C17—C20—C22	109.9 (5)
C4—C5—C6	119.9 (6)	C21—C20—C22	109.0 (5)
C4—C5—C10	115.7 (6)	C20—C22—C23	115.0 (6)
C6—C5—C10	124.3 (5)	C22—C23—C24	113.8 (6)
C5—C6—C7	124.4 (6)	C23—C24—C25	115.4 (6)
C6—C7—C8	113.0 (6)	C24—C25—C26	114.4 (8)
C7—C8—C9	110.4 (5)	C24—C25—C27	109.7 (8)
C7—C8—C14	112.1 (6)	C26—C25—C27	110.3 (7)
C9—C8—C14	110.2 (4)	O3—C28—O28	122.9 (5)
C8—C9—C10	113.4 (4)	O3—C28—C29	113.1 (4)
C8—C9—C11	111.8 (5)	O28—C28—C29	123.9 (5)
C10—C9—C11	112.8 (5)	C28—C29—C30	123.2 (5)
C1—C10—C5	108.1 (5)	C28—C29—C34	118.6 (4)
C1—C10—C9	109.3 (4)	C30—C29—C34	118.2 (4)
C1—C10—C19	110.0 (6)	C29—C30—C31	120.8 (5)
C5—C10—C9	109.9 (5)	C30—C31—C32	120.6 (4)
C5—C10—C19	108.6 (4)	O32—C32—C31	116.8 (4)
C9—C10—C19	110.8 (5)	O32—C32—C33	124.0 (5)
C9—C11—C12	114.7 (6)	C31—C32—C33	119.3 (5)
C11—C12—C13	110.9 (5)	C32—C33—C34	119.4 (5)
C12—C13—C14	105.4 (5)	C29—C34—C33	121.7 (4)
C12—C13—C17	115.9 (4)	O32—C35—C36	110.5 (4)
C12—C13—C18	110.7 (6)	C35—C36—C37	112.1 (4)
C14—C13—C17	101.4 (5)	C36—C37—C38	115.6 (4)
C14—C13—C18	112.1 (5)	C37—C38—C39	112.8 (4)
C17—C13—C18	111.0 (5)	C38—C39—C40	116.3 (4)
C8—C14—C13	115.3 (5)	C39—C40—C41	114.4 (5)
C8—C14—C15	119.0 (5)	C40—C41—C42	135 (1)

Diffraction data were processed with *Xtal*3.2 (Hall, Flack & Stewart, 1992). The phase problem was solved using the direct methods program *SIR*88 (Burla *et al.*, 1989). Non-H atoms were refined anisotropically. H atoms were located from a difference map and were refined isotropically with a fixed displacement parameter.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *Xtal*3.2 (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1989). Program(s) used to refine structure: *Xtal*3.2 *CRYLSQ*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *Xtal*3.2 *BONDLA CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: CR1163). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Crystalline Inclusion Compound and Two Host Structures Containing Chiral Molecules Derived from Lactic Acid

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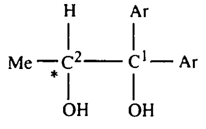
Abstract

The X-ray crystal structures of an inclusion compound with 3-picoline and two free host structures, all based on lactic acid but having different aryl substitution and optical resolution states (*S* or *RS*), are reported. They are the 2:1 complex of (*S*)-1,1-bis(*p*-*tert*-butylphenyl)-1,2-propanediol with 3-picoline, (5) [(*S*)-1,1-bis(*p*-*tert*-butylphenyl)-1,2-propanediol–3-methylpyridine (2/1), $2C_{23}H_{32}O_2.C_6H_7N$], (*S*)-1,1-bis(*p*-methylphenyl)-1,2-propanediol, (6), $C_{17}H_{20}O_2$, and (*RS*)-1,1-bis(1-naphthyl)-1,2-propanediol, (7), $C_{23}H_{20}O_2$. The main structural differences between the host molecules in the three compounds are in the conformation of the phenyl or naphthyl rings. The influence of these rings is reflected in the elongation of the C(1)—C(2) bond in the propanediol moiety [up to 1.560 (5) Å] and in the angular distortion of the tetrahedral angles around C(2). The hydroxyl groups play an important role in the crystal packing in all three. Significant differences between these structures and those of the corresponding unsubstituted phenyl host and inclusion compounds are observed.

Comment

The determination of the crystal and molecular structures of compounds (5), (6) and (7) has been undertaken as part of a broad project on crystalline inclusion compounds derived from natural lactic acid. Optically resolved crystalline hosts of type (I) have proved efficient in the enantioseparation of different guest compounds by co-crystallization or vapour sorption. The crystal structures previously studied are those of compounds (1)–(4) (Weber, Wimmer, Llamas-Saiz & Foces-Foces, 1992; Llamas-Saiz, Foces-Foces, Weber & Wimmer, 1993).



(I)

(1): Ar = Ph (2*RS*) (racemic)
(2): Ar = Ph (2*S*)
(3): (2)–3-picoline (2/1)
(4): (2)–3-methylcyclohexanone (2/1)
(5): [Ar = *p*-(*t*-BuPh) (2*S*)]–3-picoline (2/1)
(6): Ar = *p*-MePh (2*S*)
(7): Ar = 1-naphthyl (2*RS*) (racemic)

The most relevant geometrical parameters are given in Table 4 according to the atom-numbering systems shown in Fig. 1. All seven compounds studied show similar features, that is, a *gauche* conformation for the hydroxyl groups, an angular distortion of the tetrahedral angles at C(1) [105.9 (2)–114.1 (2)° in (7); see Table 4] and an opening of the *ipso* angle in the phenyl derivatives as a consequence of the σ-withdrawing effect of the substituents (Domenicano & Vaciago, 1979). The naphthalene moiety displays the same pattern of bond distances and angles as naphthalene itself (Brock & Dunitz, 1982).

The packing of (5) presents a pattern of hydrogen bonds similar to (3). Both hydroxy groups of molecule *B* are involved in strong hydrogen bonds which link the two independent molecules *A* and *B* (both with the configuration *S*) and the 3-picoline molecule. H(5)*A* is involved in an intermolecular hydrogen bond, which results in chains along the *c* axis, while the interaction of H(4)*A* is weaker than the corresponding interaction in (3) [3.543 (4) in (5) *versus* 2.805 (9) Å in (3); see Table 5 and Fig. 1*a*] probably as a result of the bulky substituents. The chains of host–guest complexes along the *c* axis are connected by methyl–phenyl interactions with the methyl H atoms pointing towards the phenyl rings. There is a sinusoidal appearance of the phenyl-ring packing along **b** and the *tert*-butyl group packing along **a** (Fig. 2). This resembles the packing in (3) but with the phenyl–phenyl interactions replaced by methyl–phenyl interactions.

In (6), the two independent molecules and those related by a twofold axis parallel to **c** are linked to form tetramers (Table 5). The crystal comprises these tetrameric units, linked together by weak phenyl–phenyl *T*-type interactions (Fig. 3). H(5)*B* appears to

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