Cholesteryl Pentyl Carbonate 의 결정 및 분자구조

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Structure of Cholesteryl Pentyl Carbonate

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요 약. Cholesteryl pentyl carbonate($C_{33}H_{56}O_3$)의 분자 및 결정구조를 X-선 회절법으로 연구하였다. 이 결정은 단사정계에 속하며 공간군은 P2$_1$이며, 단위세포 상수는 $a=12.484(3)$, $b=9.043(3)$, $c=14.053(3)$ Å, $\beta=94.12(2)°$이다. Four-circle automatic diffractometer로 회절 강도들을 얻었으며, 구조는 cholesteryl octanoate의 원자좌표를 시행구조로 이용 결정하였고 최소자승법으로 정밀화하였다. 최종 신뢰도 R 값은 1164개의 반점들에 대하여 0.12이었다. Cholesteryl 부분의 결합길이와 결합각은 정상적이나 pentyl 기는 이 영역에서의 열적 진동 때문에 결합길이가 짧았다. b-축을 따르는 2$_1$ screw symmetry에 의해 관련된 인접한 분자들은 서로 반대로 나란히 놓여 있으며 monolayer를 형성하고 있다. Monolayer 내에서 cholesteryl 군들이 서로 촘촘히 쌓인 구조를 만들어 Monolayer Type II packing mode에 해당하며, cholesteryl hexanoate, octonoate, hexyl carbonate, oleate 등의 결정구조들과 비슷한 구조로 되어 있다.

ABSTRACT. Cholesteryl pentyl carbonate ($C_{33}H_{56}O_3$) is monoclinic, space group P2$_1$, with $a=12.484(3)$, $b=9.043(3)$, $c=14.053(3)$ Å, $\beta=94.12(2)°$ and $z=2$. The intensity data were measured for the 2969 reflections within sin $\theta / \lambda = 0.52$ Å^{-1}, using an automatic four-circle diffractometer and graphite monochromated Mo–K$_\alpha$ radiation. The atomic coordinates from cholesteryl octanoate were used in an initial trial structure and the structure was refined by full-matrix least squares methods. The final R-factor was 0.12 for 1164 observed reflections. The pentyl group has shortened bond lengths due to the high thermal vibrations in this region. Adjacent molecules are related by 2$_1$ screw axis so that they are arranged in an antiparallel array, corresponding to the Monolayer Type II packing mode. There are close packings of cholesteryl groups within the monolayers. This packing type is similar to those of cholesteryl hexanoate, octanoate, hexyl carbonate and oleate.

INTRODUCTION

We have undertaken a series of crystal structures of the fatty acids esters and carbonates of cholesterol.[1-16] The crystal structures of cholesteryl pentyl (amyl) and hexyl carbonates,[2] cholesteryl formate,[3] cholesteryl chloroformate, cholesteryl isobutyrate,[5] saturated esters with chain lengths C_6–C_8[6-8] and a number of longer chain cholesteryl alkenoates have the same crystal

Table 1. Crystal data for monolayer type II cholesterol derivatives

Compound	Space Group	Unit cell Parameters
*Cholesteryl formate[3]	P2$_1$	$a = 15.757(1)$, $b = 6.073$ (1),
$C_{28}H_{46}O_2$	$z = 2$	$c = 13.592(2)$ Å, $\beta = 94.1(1)°$
*Cholesteryl chloroformate[4]	P2$_1$	$a = 12.836(2)$, $b = 9.417(2)$,
$C_{28}H_{45}ClO_2$	$z = 2$	$c = 12.327(2)$ Å, $\beta = 113.5(1)°$
*Cholesteryl isobutyrate[5]	P2$_1$	$a = 15.115(8)$, $b = 9.636(5)$,
$C_{31}H_{52}O_2$	$z = 4$	$c = 20.224(9)$ Å, $\beta = 93.15(5)°$
*+Cholesteryl hexanoate[6]	P2$_1$	$a = 12.162(3)$, $b = 9.314(3)$,
$C_{33}H_{56}O_2$	$z = 2$	$c = 13.643(5)$ Å, $\beta = 93.55(3)°$
+Cholesteryl heptanoate[7]	P2$_1$	$a = 12.54$, $b = 9.23$,
$C_{34}H_{58}O_2$	$z = 2$	$c = 14.02$ Å, $\beta = 92.0°$
*+Cholesteryl octanoate[8]	P2$_1$	$a = 12.80(3)$, $b = 9.20(2)$
$C_{35}H_{60}O_2$	$z = 2$	$c = 14.12(3)$ Å, $\beta = 93.81(3)°$
*+Cholesteryl oleate[9]	P2$_1$	$a = 12.65(3)$, $b = 9.13(3)$,
$C_{45}H_{78}O_2$	$z = 2$	$c = 18.79(5)$ Å, $\beta = 93.3(3)°$
*+Cholesteryl-*trans*-9-*trans*-12-	P2$_1$	$a = 13.03(3)$, $b = 8.76(2)$,
octadecadienoate (linolelaidate)[10]	$z = 2$	$c = 17.90(4)$ Å, $\beta = 89.7(2)°$
$C_{45}H_{76}O_2$		
+Cholesteryl *trans*-9-octadecenoate	P2$_1$	$a = 12.9(3)$, $b = 9.2(1)$,
(elaidate)[11]	$z = 2$	$c = 18.7(4)$ Å, $\beta = 93(1)°$
$C_{45}H_{78}O_2$		
+Cholesteryl *cis*-11-eicosenoate[11]	P2$_1$	$a = 12.6(2)$, $b = 9.1(1)$,
$C_{47}H_{82}O_2$	$z = 2$	$c = 19.8(4)$ Å, $\beta = 92(1)°$
+Cholesteryl *cis*-13-docosenoate	P2$_1$	$a = 13.0(3)$, $b = 9.2(1)$,
(erucate)[11]	$z = 2$	$c = 20.9(3)$ Å, $\beta = 92(1)°$
$C_{49}H_{86}O_2$		
*+Cholesteryl pentyl carbonate	P2$_1$	$a = 12.484(3)$, $b = 9.043(3)$,
$C_{33}H_{56}O_3$	$z = 2$	$c = 14.053(3)$ Å, $\beta = 94.17(2)°$
*+Cholesteryl hexyl carbonate[2]	P2$_1$	$a = 12.728(2)$, $b = 9.184(1)$,
$C_{34}H_{58}O_3$	$z = 2$	$c = 13.991(2)$ Å, $\beta = 92.93(1)°$

Structure determinations are complete for those marked(*); isostructural with that of cholesteryl octanoate is marked(+).

structure type, which have been designated Type II monolayer in order to distinguish it from other structure types.[12-16] The crystal data for these molecules are tabulated in *Table* 1.

These structures are remarkable in forming layer structures in which the central region of the layers, composed largely of semi-rigid cholesteryl groups, is closely packed and the packing of the flexible fatty acid or carbonate chains and the isoprenoid substituents at cholesterol C(17) form the interface region between layers. Within the monolayers of Type II, there is little change from one crystal structure to another.

We have determined the crystal structure of cholesteryl pentyl carbonates in order to make detailed comparisons with other type II structures.

EXPERIMENTAL

Cholesteryl pentyl carbonate from Tokyo Kasei Kogyo Co., Ltd., was crystallized by slow evaporation of an acetone solution at room temperature in the form of needles. Accurate cell parameters were determined by least-squares fit of 23 reflections with $27° < 2\theta < 35°$ using a diffractometer.

X-ray data collection was carried out using a Nonius CAD-4 diffractometer and graphite-monochromated Mo-K$_\alpha$ radiation ($\lambda = 0.7107$ Å)

by the $\omega/2\theta$ scan techniques. Three standard reflections were monitored, no significant loss of intensities being observed throughout data collection. The 2969 independent reflections were measured in range of hkl: $0 \le h \le 14$, $0 \le k \le 10$, $-16 \le l \le 16$, within $(\sin\theta)/\lambda < 0.52$ Å^{-1}.

All of the crystal data are as follows: CH_3 $(CH_2)_4OCOOC_{27}H_{45}$; Mol. Wt. 500.8; Space Group $P2_1$; $a = 12.484(3)$, $b = 9.043(3)$, $c = 14.053(3)$ Å, $\beta = 94.17(2)°$, $z = 2$; $\mu(Mo-K_a)$: 0.34 cm^{-1}; $Dc = 1.06$ g/cm^3; $D_m = 1.03$ g/cm^3 by the floatation method using a KI aqueous solution.

Since the cell parameters for cholesteryl pentyl carbonate are similar to those for cholesteryl octanoate, the atomic positional parameters of tetracyclic ring part of latter molecule were used as a starting model in Fourier refinements. The initial R-factor was 0.33, where $R = \Sigma|\Delta|/\Sigma|F_{obs}|$ and $\Delta = |F_o| - |F_c|$. The atomic paramters of tail and alkyl carbonate chain of the molecule were located in the difference Fourier maps. But several atomic peaks were poorly resolved, and as a result, the calculated bond distances and angles involving atoms of a carbonate chain and C(17) side tail were unsatisfactory.

The Fourier syntheses were calculated, which indicated the revised configurations for atoms of C(24)...C(27) and C(31)...C(33).

After several cycles of refinements, the R-factor dropped to 0.20. At this stage, anisotropic thermal parameters were introduced for the carbon and oxygen atoms, and after 10 cycles of full matrix least-suares refinements, R-factor decreased to 0.13. Hydrogen atomic positions were also calculated from the carbon framework assuming standard geometry (C-H distance is 1.08 Å). Hydrogen atoms were included in the refinement with fixed parameters, including a uniform thermal parameter of 0.05 Å^2. After final cycle of refinement, R-value was 0.12 for the 1165 observed reflections greater than $F_o > 2\sigma(F_o)$. The weighted R-value $R_w = \Sigma(w|F_o-F_c|/\Sigma wF_o)$ was

Table 2. Fractional atomic coordinates ($\times 10^3$) and equivalent isotropic temperature factors (Å$^2 \times 10^3$)of the non hydrogen atoms with e.s.d.'s in parentheses

Atom	x	y	z	U_{eq}*
C(1)	461(2)	853(3)	784(1)	89
C(2)	371(2)	826(3)	853(1)	91
C(3)	263(2)	821(3)	797(2)	85
C(4)	263(1)	706(3)	711(2)	93
C(5)	352(1)	724(3)	651(2)	69
C(6)	338(1)	729(3)	557(1)	64
C(7)	425(1)	744(3)	493(1)	77
C(8)	536(1)	717(3)	544(2)	69
C(9)	551(1)	798(3)	635(1)	71
C(10)	464(2)	734(0)	709(1)	69
C(11)	665(1)	800(3)	680(1)	72
C(12)	748(1)	848(3)	611(1)	67
C(13)	741(1)	755(3)	516(1)	57
C(14)	624(1)	772(3)	475(1)	49
C(15)	626(1)	713(3)	377(1)	70
C(16)	738(1)	756(3)	343(1)	57
C(17)	799(1)	814(3)	437(1)	65
C(18)	768(1)	590(3)	536(1)	72
C(19)	500(1)	584(3)	746(1)	77
C(20)	924(1)	779(3)	431(1)	76
C(21)	986(1)	837(3)	523(1)	98
C(22)	966(2)	847(3)	342(2)	94
C(23)	1073(2)	779(3)	315(1)	86
C(24)	1113(2)	852(4)	230(1)	119
C(25)	1218(3)	806(5)	194(2)	187
C(26)	1280(3)	701(6)	230(3)	233
C(27)	1243(2)	881(5)	108(2)	188
C(28)	96(2)	849(5)	851(2)	180
C(29)	-71(3)	831(7)	910(2)	285
C(30)	-136(2)	803(4)	986(2)	158
C(31)	-244(3)	824(6)	997(2)	261
C(32)	-302(2)	767(6)	1066(3)	238
C(33)	-404(3)	784(5)	1070(2)	190
O(3)	182(1)	782(3)	858(1)	124
O	33(1)	783(4)	913(1)	183
O(28)	61(2)	919(4)	784(1)	202

*Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

0.13, where $w = 1.232/(\sigma^2(F_o) + 0.001 (F_o))$. Maximum final shift to e.s.d. ratio was 0.98 and 1.31 for positional and thermal parameters, respectively. Final differenc Fourier map showed maximum and minimum peaks of 0.31 and 0.30 e Å^{-3}, respectively.

The final atomic coordinates and thermal parameters of the nohydrogen atoms are given in

Table 2. All calculations were performed with SHELX-76 program[17] on a IBM 3083 computer, and scattering factors taken from International Tables for X-ray Crystallography.[18]

RESULTS AND DISCUSSION

The molecular conformation of cholesteryl pentyl carbonate, which is drawn by the ORTEP program,[19] is depicted in *Fig.* 1. The bond distances and angles of cholesteryl pentyl carbonate are listed in *Table* 3. These are in agreement, within experimental error, with those found in other cholesterol derivatives. The bond distances in the tail and the pentyl carbonate show the apparent shortening which is characteristic of cholesterol derivatives, and is caused by the high thermal vibrations in these regions. In this case, it is especially pronounced in the C(25)-C(26) bond (1.31 Å) and C(32)-C(33) bond (1.29 Å).

The intramolecular distance C(3)...C(16) which is useful for comparing the length of the tetracyclic system has value of 9.00 (3)Å, which is close of the limits of range of 8.8 to 9.01 Å observed in related molecules. The torsion angle C(19)-C(10)...C(13)-C(18) which is a measure of the twist within the tetracyclic system, is 13.1 °. This torsion angle ranges between 7.9 and 18.0 ° in other related structures.

The mirror plane (ΔCs) and the two-fold (ΔC$_2$) asymmetry parameters of the ring are defined by Duax and Norton.[20]*

*ΔCs: mirror plane asymmetry parameter

$$\Delta C_s = [\sum_{i=1}^{m} (\Phi_i + \Phi_i')^2 / m]^{\frac{1}{2}}$$

ΔC$_2$: twofold asymmetry parameter

$$\Delta C_2 = [\sum_{i=1}^{m} (\Phi_i - \Phi_i')^2 / m]^{\frac{1}{2}}$$

Where ΔCs(n) is a measure of the deviations from mirror symmetry about a plane passing through atom n and the diametrically opposed atom o, and ΔC$_2$ (n-o) is a measure of the derviations from twofold symmetry about an axis bisecting bond (n-o). The symmetry related torsion angles are ϕ_i and ϕ_i', and m is the number of such pairs.
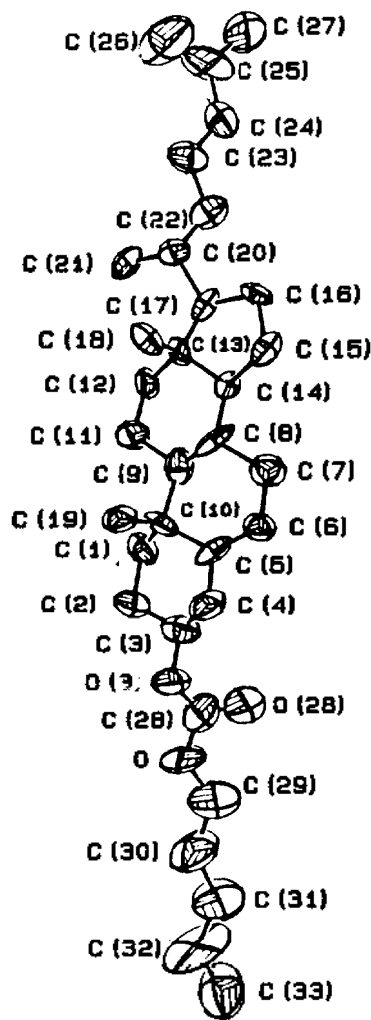

Fig. 1. Molecular conformation with atomic numbering.

The asymmetry parameters of non-ideal systems measure the degree of departure from ideal symmetry at any of the possible symmetry locations. Ring A and C assume a chair conformation: for ring A, < ΔCs > =4.6 and < ΔC$_2$ > =5.8; for ring C, < ΔCs > =4.4 and < ΔC$_2$ > =6.6. Ring B assumes a half chair and ring D the expected 13β, 14α -half chair conformations.

The torsion angle C(2)-C(3)-O(3)-C(28) which is important for determining the overall structure of the molecule is 133°, so that the carbonyl bond is parallel to the C(3)-H bond. The corresponding

Table 3. Bond lengths (Å) and angles (°) for Cholesteryl Pentyl Carbonate. The e.s.d.'s are in parentheses

C(1)-C(2)	1.56 (3)	C(1)-C(10)	1.51 (3)
C(2)-C(3)	1.50 (3)	C(3)-C(4)	1.60 (4)
C(3)-C(3)	1.42 (3)	C(4)-C(5)	1.45 (3)
C(5)-C(6)	1.32 (3)	C(5)-C(10)	1.57 (3)
C(6)-C(7)	1.47 (3)	C(7)-C(8)	1.54 (2)
C(8)-C(9)	1.47 (3)	C(8)-C(14)	1.59 (2)
C(9)-C(10)	1.67 (2)	C(9)-C(11)	1.51 (2)
C(10)-C(19)	1.51 (3)	C(11)-C(12)	1.53 (2)
C(12)-C(13)	1.57 (3)	C(13)-C(14)	1.54 (2)
C(13)-C(17)	1.47 (3)	C(13)-C(18)	1.55 (4)
C(14)-C(15)	1.48 (3)	C(15)-C(16)	1.56 (2)
C(16)-C(17)	1.56 (3)	C(17)-C(20)	1.60 (2)
C(20)-C(21)	1.54 (3)	C(20)-C(22)	1.52 (3)
C(22)-C(23)	1.55 (3)	C(23)-C(24)	1.48 (3)
C(24)-C(25)	1.50 (4)	C(25)-C(26)	1.31 (6)
C(25)-C(27)	1.43 (5)	C(28)-O(3)	1.23 (4)
C(28)-O	1.35 (4)	C(28)-O(28)	1.19 (4)
C(29)-C(30)	1.41 (4)	C(29)-O	1.36 (4)
C(30)-C(31)	1.39 (4)	C(31)-C(32)	1.34 (5)
C(32)-C(33)	1.29 (5)		
C(1)-C(2)-C(3)	110 (2)	C(2)-C(3)-C(4)	112 (2)
C(3)-C(4)-C(5)	114 (2)	C(1)-C(10)-C(5)	110 (2)
C(4)-C(5)-C(6)	122 (2)	C(5)-C(6)-C(7)	125 (2)
C(6)-C(7)-C(8)	112 (2)	C(7)-C(8)-C(9)	112 (2)
C(1)-C(10)-C(9)	104 (1)	C(5)-C(10)-C(9)	107 (1)
C(2)-C(1)-C(10)	112 (2)	C(4)-C(5)-C(10)	113 (2)
C(6)-C(5)-C(10)	124 (2)	C(8)-C(9)-C(10)	109 (2)
C(8)-C(9)-C(11)	115 (2)	C(10)-C(9)-C(11)	112 (1)
C(9)-C(11)-C(12)	113 (1)	C(7)-C(8)-C(14)	108 (2)
C(8)-C(14)-C(13)	115 (1)	C(13)-C(14)-C(15)	104 (1)
C(9)-C(8)-C(14)	109 (2)	C(13)-C(17)-C(16)	107 (2)
C(8)-C(14)-C(15)	121 (2)	C(14)-C(13)-C(17)	101 (1)
C(14)-C(15)-C(16)	106 (1)	C(12)-C(13)-C(18)	111 (2)
C(12)-C(13)-C(17)	117 (2)	C(17)-C(13)-C(18)	112 (2)
C(15)-C(16)-C(17)	102 (1)	C(5)-C(10)-C(19)	111 (1)
C(14)-C(13)-C(18)	110 (2)	C(13)-C(17)-C(20)	120 (2)
C(1)-C(10)-C(19)	115 (2)	C(17)-C(20)-C(21)	109 (2)
C(9)-C(10)-C(19)	110 (1)	C(21)-C(20)-C(22)	111 (2)
C(16)-C(17)-C(20)	108 (2)	C(22)-C(23)-C(24)	112 (2)
C(17)-C(20)-C(22)	111 (2)	C(24)-C(25)-C(26)	125 (3)
C(20)-C(22)-C(23)	114 (2)	C(26)-C(25)-C(27)	121 (3)
C(23)-C(24)-C(25)	120 (3)	C(23)-O-C(29)	116 (3)
C(24)-C(25)-C(27)	114 (2)	C(30)-C(31)-C(32)	127 (4)
C(3)-O(3)-C(28)	119 (2)	C(2)-C(3)-C(32)	127 (4)
C(29)-C(30)-C(31)	133 (3)	O-C(28)-O(3)	106 (3)
C(31)-C(32)-C(33)	124 (4)	O(3)-C(28)-O(28)	126 (3)
O(3)-C(3)-C(4)	110 (2)		
O-C(29)-C(30)	121 (3)		
O(28)-C(28)-O	124 (2)		

Table 4. Selected torsion angles (°) in Cholesteryl Pentyl Carbonate. The e.s.d.'s are in parentheses

C(1)-C(2)-C(3)-O(3)	175 (3)
C(3)-C(3)-C(4)-C(5)	-173 (3)
C(2)-C(3)-O(3)-C(28)	137 (3)
C(4)-C(3)-O(3)-O(28)	-100 (3)
O-C(28)-O(3)-C(3)	176 (4)
O(28)-C(28)-O(3)-C(3)	19 (3)
O(3)-C(28)-O-C(29)	-174 (4)
O(28)-C(28)-O-C(29)	-16 (4)
O-C(29)-C(30)-C(31)	-172 (7)
C(30)-C(29)-O-C(28)	-162 (5)
C(29)-C(30)-C(31)-C(32)	166 (7)
C(30)-C(31)-C(32)-C(33)	-176 (7)
C(13)-C(17)-C(20)-C(21)	-59 (2)
C(13)-C(17)-C(20)-C(22)	179 (3)
C(16)-C(17)-C(20)-C(21)	179 (2)
C(16)-C(17)-C(20)-C(22)	56 (2)
C(17)-C(20)-C(22)-C(23)	-162 (3)
C(21)-C(20)-C(22)-C(23)	77 (2)
C(20)-C(22)-C(23)-C(24)	-178 (3)
C(22)-C(23)-C(24)-C(25)	178 (3)
C(23)-C(24)-C(25)-C(26)	2 (3)
C(23)-C(24)-C(25)-C(27)	177 (4)

torsion angles are 121 ° in cholesteryl octanoate, 137 ° in cholesteryl oleate, 114 ° in cholesteryl hexanoate and 112 ° in cholesteryl n-hexyl carbonate.

The C(17) side chain has all trans conformation. The C(17)...C(25) distance of 6.52 Å is taken as a measure of the extension of the tail, this structure is almost fully extended. The seven atom C(17), C(20), C(22)...C(25), C(27) are in a zigzag planar chain and C(21) and C(26) are out of the plane. The conformation of the n-pentyl group also has all-trans form.

The packing diagrams are shown in Fig. 2 and 3. The planes of the cholesteryl group are parallel to ac plane with the entire molecular long axes being nearly parallel to the [20$\bar{1}$] direction. The crystal structure consists of antiparallel molecules arranged to form monolayers with a thickness of one unit cell ($d_{001} = 14.05$ Å). There is an efficient packing of cholesterol ring systems within the stacks of molecules which are related by two-fold
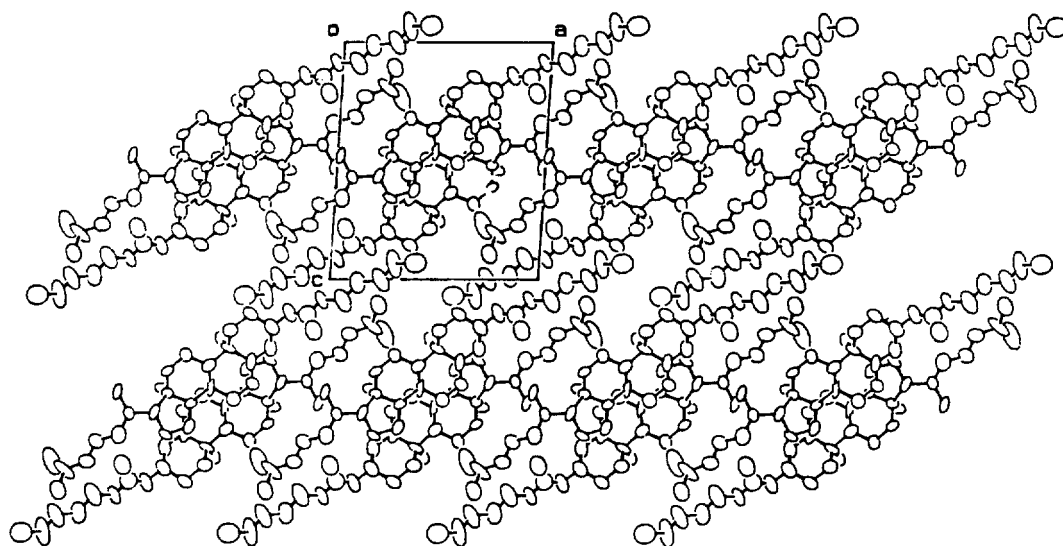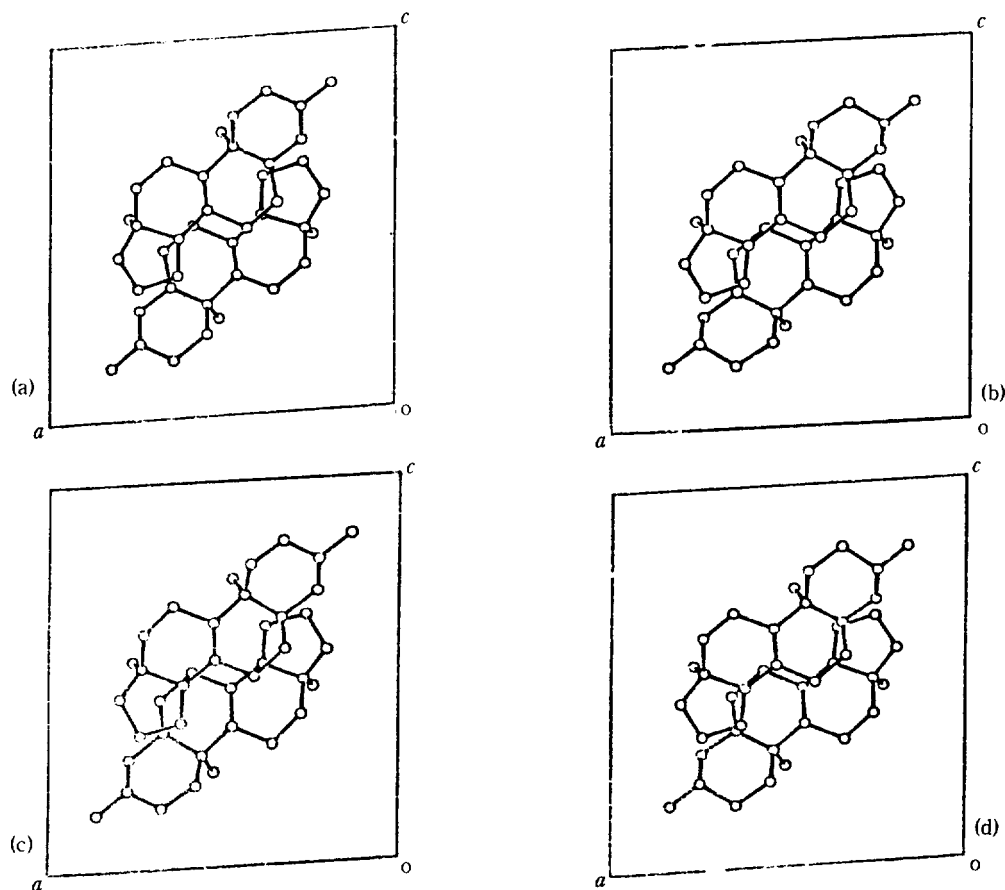
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Fig. 2. The crystal structure of cholesteryl pentyl carbonate in projection down the *b* axis. Atoms are shown as 50% probability ellipsoids.
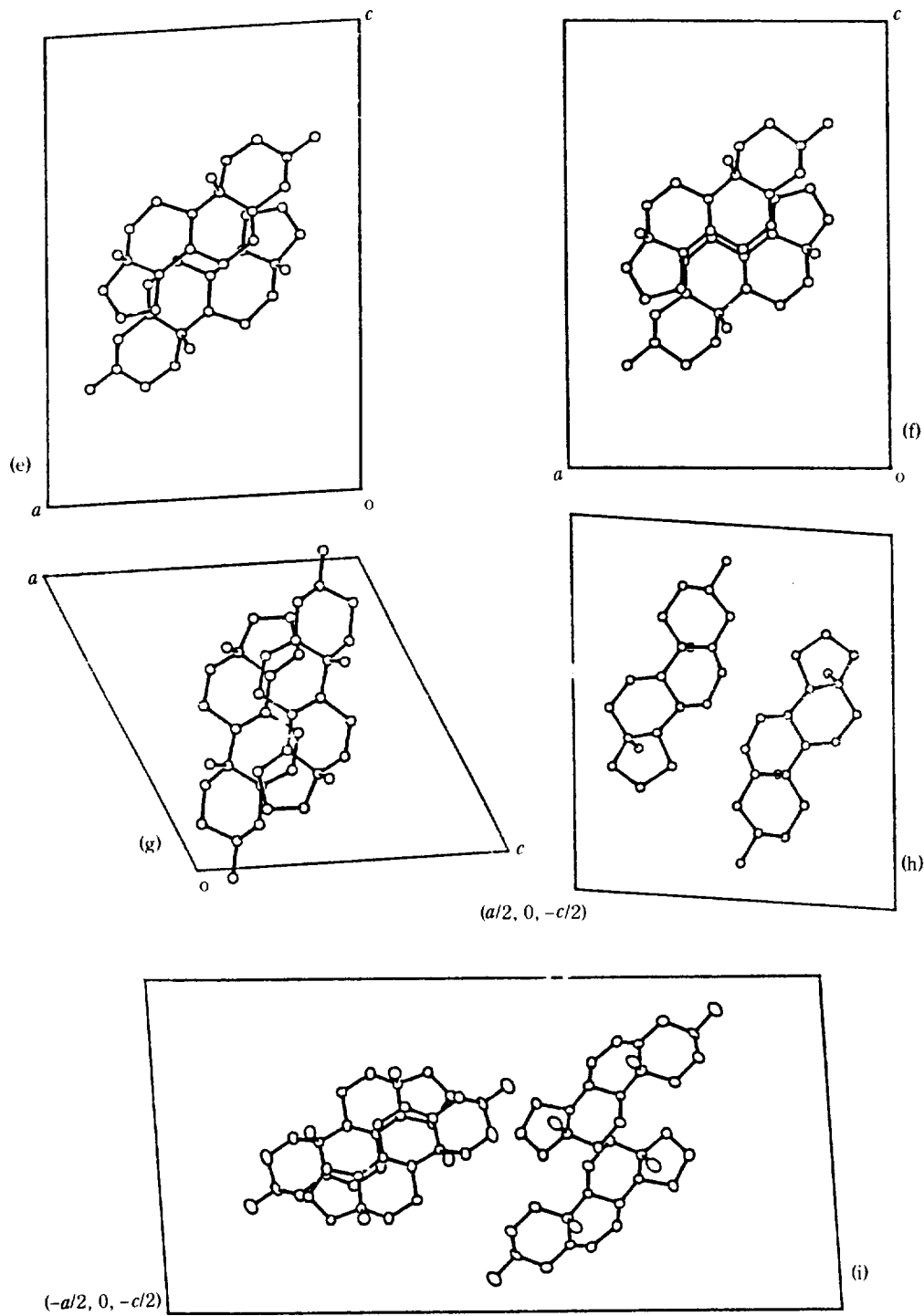
Fig. 3. The crystallographic (010) projections of the cholesterol tetracyclic ring systems of cholesteryl pentyl carbonate(a), cholesteryl hexyl carbonate(b), cholesteryl hexanoate(c), cholesteryl octanoate(d), cholesteryl oleate(e), cholesteryl linolelaidate(f), cholesteryl chloroformate(g), cholesteryl formate(h), cholesteryl isobutyrate(i).

screw axes. In directions more or less parallel to the crystal b-axis there are multiple intermolecular C...C distances less than 4.5 Å, of which the shortest (3.72 Å) is C(6)...C(18).

Similar structures of cholesteryl chloroformate, cholesteryl octanoate, hexanoate, oleate, linoledaidate and hexyl carbonate. Those are all isostructural with the others. In the six crystal structures, the arrangement within the central region of the monolayers is very similar. The difference in monolayer thickness (13.64–18.79 Å) is associated primarily with structural differences in the interlayer region. The atoms in this region consist of the alkanoate or alkyl carbonate chains of the molecules.

Crystal Structures of cholesteryl chloroformate, formate and isobutyrate also belong to monolayer type II. Those three structures are not isostructural with one another. In the cholesteryl chloroformate, there is some overlap of the B, C and D rings in a manner similar to the cholesteryl octanoate while in the formate, there is no overlap of the steroid rings viewed down the screw axis. In the structure of isobutyrate, the two distinct molecules form separated stacks with differing degrees of steroid overlap; efficient cholesteryl packing of the molecules in (A) and no overlap in molecule (B). These monolayer type II are compared as shown in *Fig.* 3.

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