& Haisa, 1989) is rather large. But in (I) and (II) the plane through C7, C1, C2, C3 makes an angle of 40·0 and 35·3°, respectively, with the phenyl ring plane C7···C12. It seems that this anomaly is caused by the benzoylamino substituent in the β position. The angle between the perfect plane 1 and the adjacent phenyl ring in (I) (19·8°) differs only slightly from that in (II) (24·0°) and the interplanar angles 1/2 in the two isomers are practically identical (43·8 and 43·7°, respectively). Short N1···O [2·574 (6) Å in (I)] and N1···S distances [3·035 (6) Å in (II)] indicate the existence of intramolecular hydrogen bonds in both isomers. The hydrogen-bond geometries normalized following Jeffrey & Lewis (1978) (N—H distance equal to 1·030 Å) are as follows: (I) H···O 2·023 (6) Å, N1—H1···O 110·7 (5)°; (II) H···S 2·294 (6) Å, N1—H1···S 127·7 (8)°. According to the compilation given in Taylor, Kennard & Versichel (1984) the hydrogen bond in (I) is a very strong one. Corresponding bond lengths in (I) and (II) agree within 3σ and are close to standard values given in literature (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). Compared with the values normally observed in cinnamic acid derivatives (see *e.g.* Bryan & Hartley, 1981; Bryan & Forcier, 1980; Iwamoto, Kashino & Haisa, 1989) two angles in both (I) and (II) show unusual features: C7—C1—C2 is reduced and C1—C2—C3 is enlarged by about 5° in each case.

The crystal structures of (I) and (II) consist of discrete molecules with all intermolecular contacts between non-H atoms greater than the sums of the corresponding van der Waals radii given by Bondi (1964).

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Structure of Chenodeoxycholic Acid in Chenodeoxycholic Acid Ethyl Acetate Solvate

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Abstract. $C_{24}H_{40}O_4.C_4H_8O_2$, $M_r = 480·7$, hexagonal, $P6_5$, $a = 22·169$ (6), $c = 10·226$ (4) Å, $V = 4352$ (2) Å^3, $Z = 6$, $D_x = 1·10$, $D_m = 1·06$ (2) g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5·7$ cm^{-1}, $F(000) = 1584$, $T = 295$ K, $R = 0·071$ for 2397 unique observed diffractometer data [$I \geq 2·5\sigma(I)$]. The chenodeoxycholic acid molecules are arranged in a helical chain around the 6_5 axis, thus forming an infinite column. The hydrophilic exteriors of these columns are stacked parallel to each other and connected by helical hydrogen-bond chains around the 2_1 axes. The mainly hydrophobic interiors of the channels, left open around the 6_5 axis, are filled with severely disordered ethyl acetate molecules. The steroid nucleus has a conformation typical for bile acids. The 17β side chain is not extended, in order to accommodate the hydrogen-bond network.

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Introduction. The bile acids are hydroxylated derivatives of the steroid 5β-cholan-24-oic acid. They play important physiological roles in the digestion and absorption of fats (Bloom & Fawcett, 1975). Deoxycholic acid (DCA, 3α,12α-dihydroxy-5β-cholan-24-oic acid) interacts strongly with organic molecules in solution. X-ray crystallographic studies were carried out on several DCA complexes to understand the physiological roles of bile acids and because of the intrinsic crystallographic interest in these molecular complexes. Most of the these DCA complexes are orthorhombic (*e.g.* Jones, Schwarzbaum, Lessinger & Low, 1982, and references therein) and have a hydrogen-bonded bilayer of DCA molecules with hydrophobic channels. Without significant conformational changes of the DCA molecules this bilayer structure accommodates a wide variety of organic compounds. Also a tetragonal complex is known (Coiro, D'Andrea & Giglio, 1979). In this ethanol–water complex the channels are hydrophilic. Two hexagonal complexes are known (Candeloro De Sanctis, Coiro, Giglio, Pagliuca, Pavel & Quagliata, 1978; Candeloro De Sanctis, Giglio, Petri & Quagliata, 1979), where the DCA molecules form hydrogen-bonded helices around the 6_5 axes and the guest molecules are hydrogen bonded to the hydrophilic channel walls. Chenodeoxycholic acid (CDCA, 3α,7α-dihydroxy-5β-cholan-24-oic acid) is capable of forming similar complexes. The structure of the solvent-free high-melting polymorph of CDCA has been reported (Lindley, Mahmoud, Watson & Jones, 1980). They also obtained small crystals of a low-melting tenaciously solvent-retaining pseudo polymorph with hexagonal symmetry, which showed signs of disorder and were unsuitable for X-ray analysis. We now report the crystal structure of the ethyl acetate solvate of CDCA, also with hexagonal symmetry.

Experimental. The title compound was obtained from Organon International and was crystallized from a 1:1 mixture of chloroform and ethyl acetate. Data were collected on a CAD4-F diffractometer for a transparent, rod-shaped colorless crystal (0·8 × 0·4 × 0·2 mm), mounted on a glass fiber. The density was obtained by flotation in an aqueous solution of potassium iodide. The cell parameters were calculated by least-squares fit from the setting angles of 15 reflections with $19 \le \theta \le 31°$. 3401 reflections were scanned [h 0:23, k 0:23, l 0:12; $\theta \le 69{\cdot}96°$; $\omega/2\theta$-scan mode; $\Delta\omega = (1{\cdot}0 + 0{\cdot}14\tan\theta)°$; Ni-filtered Cu $K\alpha$ radiation]. Two reference reflections ($\bar{5}52$, $0\bar{5}2$) showed fluctuations of 2%, and a decay of less than 7% during the 78 h of X-ray exposure time. The data were corrected for Lp but not for absorption, resulting in the unique set of 2397 observed reflections [$I > 2{\cdot}5\sigma(I)$] used in the structure determination. $\sigma^2(I) = \sigma_{cs}^2(I) + (pI)^2$ (McCandlish, Stout & Andrews, 1975) with $p = 0{\cdot}06$. All non-hydrogen atoms of the chenodeoxycholic acid molecules were found by direct methods (*SHELXS*86; Sheldrick, 1986). The structure was refined on F by full-matrix least-squares procedures using anisotropic thermal parameters for all non-hydrogen atoms (*SHELX*76; Sheldrick, 1976). The hydroxyl hydrogen atoms were located from a difference Fourier map and refined with distance restraints. All other H atoms were introduced at calculated positions (C—H = 0·98 Å) and refined in the riding mode on their carrier atoms with a general isotropic thermal parameter. Convergence was reached at $R = 0{\cdot}126$ ($wR = 0{\cdot}242$, $w = 1{\cdot}0$). A subsequent difference Fourier map showed a very large volume along the 6_5 axis (1220 Å^3; *BYPASS*, van der Sluis & Spek, 1990) with a diffuse electron density (< 0·68 e Å^{-3}). Because no solvent model could be fitted in this electron density the *BYPASS* procedure (van der Sluis & Spek, 1990) was used to take this electron density into account in the refinement. The distance restraints on the hydroxyl hydrogen atoms could be removed. Final convergence was reached at $R = 0{\cdot}071$ [$wR = 0{\cdot}068$, $w = 9{\cdot}2/\sigma^2(F)$; $S = 1{\cdot}24$; $(\Delta/\sigma)_{max} = 0{\cdot}85$; number of refined parameters = 262]. There was no residual density outside the range −0·24 to 0·37 e Å^{-3}. Scattering factors were from Cromer & Mann (1968) and anomalous-dispersion corrections from Cromer & Liberman (1970). The final atomic coordinates and

Table 1. *Final coordinates and equivalent isotropic thermal parameters, with e.s.d.'s in parentheses*

	x	y	z	U_{eq} (Å^2)†
O(3)	0·0059 (2)	0·4401 (4)	0·5598	0·066 (2)
O(7)	−0·0829 (2)	0·4141 (4)	0·0981 (6)	0·050 (1)
O(241)	−0·3331 (2)	0·0227 (5)	−0·4458 (7)	0·077 (2)
O(242)	−0·4107 (3)	−0·0381 (5)	−0·2953 (6)	0·081 (2)
C(1)	−0·1878 (3)	0·3312 (5)	0·5335 (7)	0·051 (2)
C(2)	−0·1128 (3)	0·3477 (5)	0·5560 (6)	0·050 (2)
C(3)	−0·0652 (3)	0·4252 (5)	0·5328 (7)	0·050 (2)
C(4)	−0·0710 (3)	0·4427 (5)	0·3961 (6)	0·045 (2)
C(5)	−0·1468 (2)	0·4251 (5)	0·3638 (6)	0·043 (2)
C(6)	−0·1546 (3)	0·4438 (5)	0·2253 (7)	0·049 (2)
C(7)	−0·1542 (3)	0·3984 (5)	0·1155 (7)	0·048 (2)
C(8)	−0·2032 (3)	0·3192 (6)	0·1469 (6)	0·047 (2)
C(9)	−0·1926 (3)	0·3024 (6)	0·2868 (7)	0·049 (2)
C(10)	−0·1996 (2)	0·3465 (5)	0·3945 (6)	0·043 (2)
C(11)	−0·2361 (3)	0·2226 (6)	0·3126 (6)	0·054 (2)
C(12)	−0·2335 (3)	0·1780 (7)	0·2073 (7)	0·068 (2)
C(13)	−0·2495 (3)	0·1923 (6)	0·0727 (7)	0·061 (2)
C(14)	−0·2000 (3)	0·2727 (5)	0·0459 (7)	0·050 (2)
C(15)	−0·2089 (3)	0·2813 (6)	−0·0959 (7)	0·061 (2)
C(16)	−0·2211 (3)	0·2145 (7)	−0·1613 (7)	0·072 (2)
C(17)	−0·2321 (3)	0·1642 (6)	−0·0486 (7)	0·065 (2)
C(18)	−0·3267 (3)	0·1757 (6)	0·0667 (7)	0·068 (2)
C(19)	−0·2739 (2)	0·3373 (5)	0·3895 (7)	0·054 (2)
C(20)	−0·2807 (4)	0·0857 (7)	−0·0879 (8)	0·080 (3)
C(21)	−0·3022 (4)	0·0348 (8)	0·027 (1)	0·106 (4)
C(22)	−0·2477 (3)	0·0666 (7)	−0·1975 (9)	0·088 (3)
C(23)	−0·2914 (4)	−0·0042 (7)	−0·2585 (8)	0·087 (3)
C(24)	−0·3508 (4)	−0·0066 (8)	−0·3324 (7)	0·067 (3)

†$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

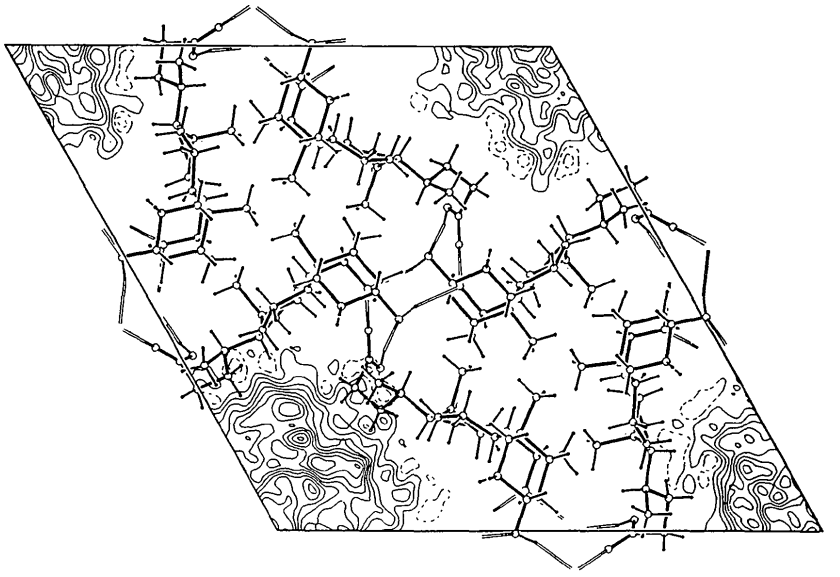

Fig. 1. Projection of the structure down the c axis, superimposed at $z = 0{\cdot}11$ of the Fourier synthesis calculated from the continuous solvent contribution to F_c. Contour levels are at $0{\cdot}10$ e Å^{-3}, with the minimum $-0{\cdot}15$ and maximum $0{\cdot}77$ e Å^{-3}. Hydrogen bonds are indicated with open lines.

equivalent isotropic temperature factors are listed in Table 1.* The program package *EUCLID* (Spek, 1982) was used for geometrical calculations and illustrations. All calculations were carried out on a MicroVAX II.

Discussion. The hexagonal unit cell contains six molecules of CDCA and one solvent channel as shown in Fig. 1. The molecular structure with labeling is depicted in Fig. 2. Bond distances and angles and hydrogen-bond geometry are listed in Table 2.

Molecular conformation. The conformation of the molecule shows no unexpected features and differs mainly in the values of the torsion angles around C(20)—C(22)—C(23)—C(24) [68·3 (10)°] and C(22)—C(23)—C(24)—O(241) [79·1 (9)°] when compared with the two independent molecules in the unsolvated modification of CDCA (65 and 179, 170 and −179°, respectively). This conformational flexibility in the 17β side chain is also found for other cholane compounds and is dominated by intermolecular hydrogen bonding. According to Arora, Germain & Declercq (1976) an antiperiplanar conformation around the C(20)—C(22) bond indicates a lack of hydrogen bonding for the carboxyl group. Thus in lithocholic acid (Arora, Germain & Declercq, 1976) and DCA (Candeloro De Sanctis, Giglio, Pavel & Quagliata, 1972) where the C(17)—C(20)—C(22)—C(23) torsion angles are 64 and 62° respectively the carboxyl groups are involved in hydrogen bonding, but not in 3α,6α-dihydroxy-5β-cholan-24-oic acid (Hall, Maslen & Cooper, 1974), 4′-bromo-3α,12α-dihydroxy-5β-cholan-24-anilide (Schaefer & Reed, 1972) and cholic acid (Johnson & Schaefer, 1972), where the conformation is antiperiplanar (mean −175°). CDCA molecules in crystal structures are anomalous in this respect, having complete intermolecular hydrogen bonding with a torsion angle of −172·6 (7)°. This conformation was also found for the unsolvated form of CDCA where both crystallographically independent molecules are completely hydrogen bonded and have torsion angles −176 and 180° respectively. The shortening of the C—O bond length (from 1·450 to 1·425 Å), as observed by Arora, Germain & Declercq (1976), in the hydrogen-bonded case, is not found here. However, this shortening was indeed observed by Lindley, Mahmoud, Watson & Jones (1980) in the structure of the solvent-free CDCA.

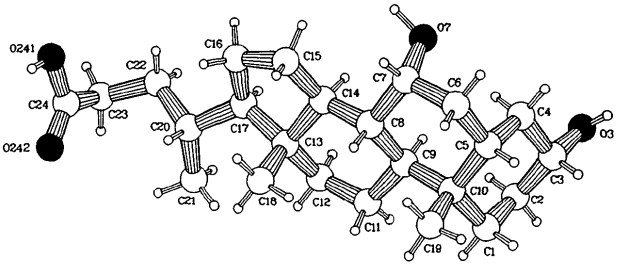

Fig. 2. *PLUTON* (Spek, 1982) drawing of the molecular structure with adopted labeling.

* Lists of anisotropic thermal parameters, hydrogen-atom positions, bond angles and distances involving hydrogen atoms, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53007 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *bond angles* (°) *and hydrogen-bond geometry* (Å *and* °) *with e.s.d.'s in parentheses*

O(3)—C(3)	1·466 (9)	C(9)—C(11)	1·56 (1)
O(7)—C(7)	1·45 (1)	C(10)—C(19)	1·554 (9)
O(241)—C(24)	1·29 (1)	C(11)—C(12)	1·48 (1)
O(242)—C(24)	1·21 (1)	C(12)—C(13)	1·49 (1)
C(1)—C(2)	1·53 (1)	C(13)—C(14)	1·58 (1)
C(1)—C(10)	1·51 (1)	C(13)—C(17)	1·52 (1)
C(2)—C(3)	1·52 (1)	C(13)—C(18)	1·56 (1)
C(3)—C(4)	1·47 (1)	C(14)—C(15)	1·49 (1)
C(4)—C(5)	1·56 (1)	C(15)—C(16)	1·52 (1)
C(5)—C(6)	1·51 (1)	C(16)—C(17)	1·54 (1)
C(5)—C(10)	1·57 (1)	C(17)—C(20)	1·57 (1)
C(6)—C(7)	1·51 (1)	C(20)—C(21)	1·53 (1)
C(7)—C(8)	1·57 (1)	C(20)—C(22)	1·51 (1)
C(8)—C(9)	1·53 (1)	C(22)—C(23)	1·51 (1)
C(8)—C(14)	1·49 (1)	C(23)—C(24)	1·50 (1)
C(9)—C(10)	1·53 (1)		

C(2)—C(1)—C(10)	112·2 (6)	C(9)—C(10)—C(19)	109·4 (6)
C(1)—C(2)—C(3)	107·7 (6)	C(11)—C(12)—C(13)	115·8 (7)
O(3)—C(3)—C(2)	106·6 (6)	C(12)—C(13)—C(14)	107·1 (6)
O(3)—C(3)—C(4)	110·2 (6)	C(12)—C(13)—C(17)	121·9 (7)
C(2)—C(3)—C(4)	110·1 (6)	C(12)—C(13)—C(18)	109·5 (6)
C(3)—C(4)—C(5)	111·1 (6)	C(14)—C(13)—C(17)	98·3 (6)
C(4)—C(5)—C(6)	112·8 (6)	C(14)—C(13)—C(18)	108·7 (6)
C(4)—C(5)—C(10)	110·4 (5)	C(17)—C(13)—C(18)	110·1 (6)
C(6)—C(5)—C(10)	111·6 (6)	C(8)—C(14)—C(13)	114·5 (6)
C(5)—C(6)—C(7)	118·3 (6)	C(8)—C(14)—C(15)	122·2 (7)
O(7)—C(7)—C(6)	107·5 (6)	C(13)—C(14)—C(15)	104·6 (6)
O(7)—C(7)—C(8)	110·8 (6)	C(14)—C(15)—C(16)	105·9 (6)
C(6)—C(7)—C(8)	111·2 (6)	C(15)—C(16)—C(17)	105·3 (6)
C(7)—C(8)—C(9)	111·1 (6)	C(13)—C(17)—C(16)	107·2 (7)
C(7)—C(8)—C(14)	112·8 (6)	C(13)—C(17)—C(20)	119·2 (7)
C(9)—C(8)—C(14)	114·6 (6)	C(16)—C(17)—C(20)	112·6 (6)
C(8)—C(9)—C(10)	116·2 (6)	C(17)—C(20)—C(21)	114·1 (7)
C(8)—C(9)—C(11)	110·7 (6)	C(17)—C(20)—C(22)	109·9 (8)
C(10)—C(9)—C(11)	113·6 (6)	C(21)—C(20)—C(22)	112·2 (8)
C(1)—C(10)—C(5)	108·6 (6)	C(20)—C(22)—C(23)	116·7 (8)
C(1)—C(10)—C(9)	116·5 (6)	C(22)—C(23)—C(24)	110·0 (8)
C(1)—C(10)—C(19)	107·0 (6)	O(241)—C(24)—O(242)	122·8 (8)
C(5)—C(10)—C(9)	107·8 (5)	O(241)—C(24)—C(23)	113·3 (8)
C(5)—C(10)—C(19)	107·1 (6)	O(242)—C(24)—C(23)	123·6 (8)
C(9)—C(11)—C(12)	115·2 (6)		

D—H···*A*	*D*···*A*	*D*—H	H···*A*	*D*—H···*A*
O(3)—H(32)···O(7)^{i}	2·828 (8)	0·89 (8)	1·96 (8)	165 (7)
O(7)—H(72)···O(242)^{ii}	2·850 (9)	1·00 (6)	2·01 (7)	140 (6)
O(241)—H(241)···O(3)^{iii}	2·654 (9)	0·75 (8)	1·97 (7)	152 (7)

Symmetry code: (i) $-x, -y+1, \frac{1}{2}+z$; (ii) $y, -x+y, \frac{1}{6}+z$; (iii) $x-y, x, -\frac{7}{6}+z$.

Hydrogen bonding. The CDCA molecules are held together by an extensive hydrogen-bond network involving all available hydrogen-bond donors (Table 2; Fig. 1). Around the 2_1 axis an infinite hydrogen-bond chain O(241)—H(241)···O(3)—H(32)···O(7)—H(72)···O(242)—C(24)—O(241)′, where O(241)′ is related to O(241) by the 2_1 operation, is found. These chains occur at both ends of all molecules and therefore result in a three-dimensional hydrogen-bond network. The packing leaves a large hydrophobic channel open around the 6_5 axis (1220 Å^3, *i.e.* 28% of the total volume). Such a large solvent channel was also observed for both hexagonal phases of DCA (Candeloro De Sanctis, Coiro, Giglio, Pagliuca, Pavel & Quagliata, 1978; Candeloro De Sanctis, Giglio, Petri & Quagliata, 1979).

Continuous electron density channel. Ethyl acetate was used in the crystallization of the title compound. The electron count in the solvent channel, as obtained with the *BYPASS* procedure (van der Sluis & Spek, 1989) yielded 294 electrons, equivalent to with 1·0 ethyl acetate molecules in the asymmetric unit. The corresponding calculated density of 1·10 Mg m^{-3} is in agreement with the experimental D_m = 1·06 (2) Mg m^{-3}. The hydrophobic nature of the channel wall may be the cause of the disordering of the solvent molecules.

Note added in proof: Very recently, Rizkallah, Harding, Lindley, Aigner & Bauer (1990) presented a structure of a pseudo polymorph of CDCA, crystallized from chloroform. Structure determination of only moderate quality was possible (R = 0·11 for only 578 reflections) even with a synchrotron radiation source and a Fast diffractometer. Apart from the disordered solvent both crystals were isostructural.

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