Organic Chemistry, Section E (IUPAC, 1976) (*i.e.* head-to-tail *cis-anti-cis* dimer) possesses a '*Z* structure' compared to the '*U* structure' of the htt *cis-syn-cis* dimer (El Amoudi El Faghi *et al.*, 1987).

References

El Amoudi El Faghi, M. S., Geneste, P. & Olivé, J. L. (1981). *Nouv. J. Chim.* **5**, 251–255.

El Amoudi El Faghi, M. S., Geneste, P., Olivé, J. L., Rambaud, J. & Declercq, J.-P. (1987). *Acta Cryst.* C**43**, 561–562.

El Amoudi El Faghi, M. S., Geneste, P., Olivé, J. L., Rambaud, J. & Declercq, J.-P. (1988). *Acta Cryst.* C**44**, 498–500.

Hopkinson, M. J., Schloman, W. W., Plummer, B. F., Wenkert, E. & Raju, M. (1979). *J. Am. Chem. Soc.* **101**, 2157–2164.

IUPAC (1976). *Pure Appl. Chem.* **45**, 11–30.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*78. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Roberts, P. & Sheldrick, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1992). C**48**, 503–507

Structures of 1:1 Addition Compounds of Methyl Cholate with Methanol and with 2-Propanol

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Abstract. (1): Methyl cholate–methanol (1/1), $C_{25}H_{42}O_5.CH_4O$, $M_r = 454.65$, monoclinic, $C2$, $a = 25.184\,(3)$, $b = 7.797\,(1)$, $c = 15.174\,(3)$ Å, $\beta = 121.05\,(1)^\circ$, $U = 2552.8\,(7)$ Å^3, $Z = 4$, $D_x = 1.183$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 6.23$ cm^{-1}, $F(000) = 1000$, $T = 295$ K, $R = 0.033$ for 1844 observed reflections. (2): Methyl cholate–2-propanol (1/1), $C_{25}H_{42}O_5.C_3H_8O$, $M_r = 482.70$, monoclinic, $C2$, $a = 25.835\,(2)$, $b = 8.137\,(1)$, $c = 15.553\,(2)$ Å, $\beta = 121.08\,(1)^\circ$, $U = 2800.3\,(3)$ Å^3, $Z = 4$, $D_x = 1.145$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 5.92$ cm^{-1}, $F(000) = 1064$, $T = 295$ K, $R = 0.067$ for 1944 observed reflections. The molar ratio between methyl cholate (MC) and the guest molecules (methanol and 2-propanol) is 1:1. The crystal structures are isomorphous. The MC molecules form layered arrangements, however, which provide no hydrophobic channel spaces. Alcohol molecules are accommodated by the host steroidal molecules with hydrogen bonding.

Introduction. It is known that the typical steroidal bile acid, deoxycholic acid (3α,12α-dihydroxy-5β-cholan-24-oic acid; DCA) accommodates a variety of

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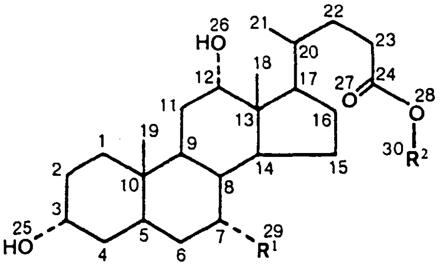
organic guest molecules in tunnel-like spaces, usually called 'channels' which are formed in the crystalline states of DCA (Giglio, 1984). We have found that these channels of DCA can also include organometallic compounds such as ferrocene (Miki, Kasai, Tsutsumi, Miyata & Takemoto, 1987). We have discovered that these channel spaces also occur when acetophenone is included by cholic acid (3α,7α,12α-trihydroxy-5β-cholan-24-oic acid; CA), which has only one additional OH group compared to DCA (Miki, Masui, Kasai, Miyata, Shibakami & Takemoto, 1988). In the crystalline state, CA permits optical resolution of racemic lactones (Miki, Kasai, Shibakami, Takemoto & Miyata, 1991) and demonstrates intercalation phenomena for organic molecules (Miyata, Shibakami, Chirachanchai, Takemoto, Kasai & Miki, 1990).

We are also interested in the crystalline states of DCA and CA esters, particularly which types of crystalline assemblies are formed by the esters of these bile acids. We have already reported the crystal structure of the addition compound of methyl deoxycholate (MDC) with methanol, where no channel spaces for the guest methanol were observed (Miki, Masui, Kasai, Miyata, Goonewardena, Shibakami & Takemoto, 1989).

Here we report the crystal structures of addition compounds of the methyl ester of CA, methyl cholate (MC) with methanol (1) and 2-propanol (2). The structure of (1) has been partly described previously (Miyata, Goonewardena, Shibakami, Takemoto, Masui, Miki & Kasai, 1987).



Deoxycholic acid (DCA)	R^1 = H,	R^2 = H
Cholic acid (CA)	R^1 = OH,	R^2 = H
Methyl deoxycholate (MDC)	R^1 = H,	R^2 = CH_3
Methyl cholate (MC)	R^1 = OH,	R^2 = CH_3

Experimental. Crystals of the title compounds, (1) and (2), were grown as colorless prisms from methanol and 2-propanol solutions of MC, respectively. Well-shaped crystals with dimensions of 0.25 × 0.25 × 0.50 mm (1) and 0.25 × 0.25 × 0.30 mm (2) were mounted on a Rigaku automated four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation. Unit-cell parameters were determined by least-squares fit of 2θ values of 25 reflections in the range 33.2–69.3°. Intensities were measured by the θ–2θ scan technique with a scan rate of 4° $\min^{-1}$ in 2θ and with a scan width of $\Delta(2\theta) = (sw + 0.70\tan\theta)^\circ$, where sw = 2.0 and 1.6 for (1) and (2), respectively. Background intensities were measured for 7.5 s (1) and 5 s (2) at each end of a scan. Four standard reflections [0$\bar{4}$0, 0$\bar{2}$4, 800, 004 for (1) and 240, 060, 10,0,0, 006 for (2)] were re-measured every 60 reflections, no significant loss in intensities being observed. Totals of 1934 and 2112 independent reflections were collected with 2θ up to 116.0° ($\sin\theta/\lambda = 0.550$ Å^{-1}) and index ranges of $h = 0$ to 27, $k = -8$ to 0, $l = -16$ to 14 for (1) and $h = -28$ to 24, $k = 0$ to 8, $l = 0$ to 17 for (2). Lp corrections were applied, but neither absorption nor extinction corrections were carried out.

The structure of (1) was solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The initial E map gave a partial structure of the MC molecule, including 21 of the 32 non-H atoms. The positions of the remaining non-H atoms were located from the subsequent Fourier synthesis. In addition, the atomic positions of the MC molecule in structure (1) were employed for the initial model of (2), because of the isomorphism between crystals (1) and (2). After several cycles of refinement of this initial model, the atomic positions of four non-H atoms of 2-propanol were found from a difference Fourier map.

The structure was refined by the block-diagonal least-squares procedure using the program *HBLS*-V (Ashida, 1979). Reflections with $|F_o| \geq 3\sigma(|F_o|)$ were included in the refinement, where the function minimized was $\sum w(|F_o| - |F_c|)^2$. On the difference Fourier maps all the H atoms were found at the expected positions. Their parameters were further refined except for those of the methyl H atoms of 2-propanol in (2). The weighting scheme used was $w = [\sigma(F_o)^2 + a|F_o| + b|F_o|^2]^{-1}$, where a and b used in the final refinement are 0.0250 and 0.0003 for (1) and 0.1200 and 0.0005 for (2). Several strong reflections [110, 400, $\bar{1}$11, $\bar{5}$13 for (1) and 110, 400, $\bar{1}$11, 001, $\bar{6}$03, $\bar{5}$13 for (2)], which were considered to be affected by extinction, were omitted in the final stages of refinement. The number of observations (m) per refined parameters (s) is 1844/474 = 3.89 with S = 1.1 for (1), and 1944/484 = 4.02 with S = 1.1 for (2). The final R and wR values for (1) are 0.033 and 0.043 for 1844 reflections, while those for (2) are 0.067 and 0.093 for 1944 reflections. The relatively high R values for (2) are probably due to the large thermal motions of the guest 2-propanol molecule. The $(\Delta/\sigma)_{\max}$ values of non-H atoms in the final refinement cycle are 0.19 and 0.31 for (1) and (2), respectively. The peaks in the final $\Delta\rho$ map were between 0.06 and -0.07 e Å^{-3} for (1) and between 0.31 and -0.10 e Å^{-3} for (2). The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final atomic parameters for (1) and (2) are listed in Table 1.*

All computations were performed on ACOS 850 and ACOS 930 computers at the Research Center for Protein Engineering (formerly Crystallographic Research Center), Institute for Protein Research, Osaka University.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters of non-*H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*(Å^2)
MC–methanol (1)				
C(1)	0.15133 (13)	−0.2027 (5)	0.0974 (2)	3.5
C(2)	0.08558 (12)	−0.1907 (4)	0.0034 (2)	3.4
C(3)	0.07802 (12)	−0.0202 (5)	−0.0491 (2)	3.3
C(4)	0.08991 (12)	0.1257 (4)	0.0240 (2)	3.3
C(5)	0.15493 (12)	0.1164 (5)	0.1216 (2)	3.1
C(6)	0.16517 (13)	0.2697 (5)	0.1924 (2)	3.7
C(7)	0.13431 (13)	0.2523 (4)	0.2562 (2)	3.4
C(8)	0.14931 (12)	0.0794 (4)	0.3112 (2)	2.9
C(9)	0.13271 (11)	−0.0710 (4)	0.2350 (2)	2.8
C(10)	0.16916 (11)	−0.0584 (4)	0.1774 (2)	3.0
C(11)	0.13781 (13)	−0.2472 (4)	0.2855 (2)	3.3
C(12)	0.10885 (12)	−0.2556 (4)	0.3532 (2)	3.0
C(13)	0.13406 (11)	−0.1121 (4)	0.4342 (2)	2.7
C(14)	0.11896 (11)	0.0593 (4)	0.3756 (2)	2.8
C(15)	0.13303 (14)	0.1938 (4)	0.4571 (3)	3.7
C(16)	0.11507 (14)	0.1077 (5)	0.5296 (3)	3.9
C(17)	0.10208 (11)	−0.0845 (4)	0.4975 (2)	2.9
C(18)	0.20424 (12)	−0.1374 (5)	0.5076 (2)	3.6
C(19)	0.23986 (13)	−0.0747 (6)	0.2534 (3)	4.4
C(20)	0.12063 (11)	−0.2036 (5)	0.5908 (2)	3.2
C(21)	0.10747 (15)	−0.3921 (5)	0.5596 (3)	4.3
C(22)	0.08747 (14)	−0.1517 (5)	0.6481 (3)	4.2
C(23)	0.12224 (15)	−0.2136 (5)	0.7600 (3)	4.7
C(24)	0.17669 (15)	−0.1032 (5)	0.8285 (3)	4.3
O(25)	0.01661 (9)	0.0043 (4)	−0.1358 (2)	4.2
O(26)	0.04214 (8)	−0.2394 (3)	0.2901 (2)	3.5
O(27)	0.18351 (16)	0.0437 (5)	0.8142 (2)	8.3
O(28)	0.21803 (11)	−0.1866 (4)	0.9135 (2)	5.0
O(29)	0.06824 (9)	0.2827 (3)	0.1946 (2)	3.8
C(30)	0.27111 (17)	−0.0906 (6)	0.9889 (3)	6.1
C(*M*)	0.04113 (22)	0.3370 (7)	0.7284 (5)	8.1
O(*M*)	0.00860 (15)	0.4452 (4)	0.7533 (3)	8.8
MC–2-propanol (2)				
C(1)	0.1517 (3)	−0.2027 (9)	0.0961 (5)	7.2
C(2)	0.0868 (3)	−0.1970 (8)	0.0065 (5)	6.6
C(3)	0.0744 (3)	−0.0388 (9)	−0.0500 (4)	6.1
C(4)	0.0849 (3)	0.1039 (8)	0.0197 (4)	5.7
C(5)	0.1494 (3)	0.1055 (9)	0.1108 (4)	6.3
C(6)	0.1591 (3)	0.2554 (9)	0.1769 (5)	6.8
C(7)	0.1305 (3)	0.2383 (8)	0.2414 (4)	5.9
C(8)	0.1512 (2)	0.0788 (7)	0.3024 (4)	5.2
C(9)	0.1360 (2)	−0.0716 (8)	0.2310 (4)	5.3
C(10)	0.1687 (3)	−0.0579 (9)	0.1707 (4)	6.1
C(11)	0.1458 (3)	−0.2350 (8)	0.2853 (5)	6.6
C(12)	0.1191 (3)	−0.2456 (8)	0.3539 (4)	6.0
C(13)	0.1408 (2)	−0.1017 (7)	0.4288 (4)	5.1
C(14)	0.1241 (2)	0.0572 (7)	0.3680 (4)	5.0
C(15)	0.1366 (3)	0.1935 (8)	0.4453 (4)	5.8
C(16)	0.1216 (3)	0.1093 (9)	0.5189 (4)	6.2
C(17)	0.1095 (2)	−0.0744 (8)	0.4904 (4)	5.2
C(18)	0.2097 (3)	−0.1184 (10)	0.5018 (5)	6.8
C(19)	0.2384 (3)	−0.0594 (13)	0.2410 (5)	8.2
C(20)	0.1259 (3)	−0.1873 (9)	0.5811 (4)	6.3
C(21)	0.1173 (4)	−0.3666 (10)	0.5562 (5)	8.1
C(22)	0.0904 (3)	−0.1351 (11)	0.6307 (5)	7.5
C(23)	0.1200 (4)	−0.1858 (12)	0.7410 (5)	8.5
C(24)	0.1744 (3)	−0.0847 (11)	0.8118 (5)	7.8
O(25)	0.0135 (2)	−0.0362 (6)	−0.1356 (3)	7.3
O(26)	0.0544 (2)	−0.2541 (6)	0.2963 (3)	6.5
O(27)	0.1864 (4)	0.0484 (11)	0.7968 (5)	14.2
O(28)	0.2074 (3)	−0.1601 (9)	0.8955 (4)	9.3
O(29)	0.0660 (2)	0.2455 (5)	0.1804 (3)	6.0
C(30)	0.2594 (4)	−0.0701 (16)	0.9714 (6)	10.7
C(*P*1)	0.0370 (4)	0.4115 (14)	0.7305 (7)	9.9
C(*P*2)	0.0257 (5)	0.3170 (34)	0.6514 (9)	20.8
C(*P*3)	0.0910 (5)	0.4051 (21)	0.8203 (7)	13.1
O(*P*)	−0.0136 (3)	0.4355 (7)	0.7407 (5)	9.5

* As defined by Hamilton (1959).

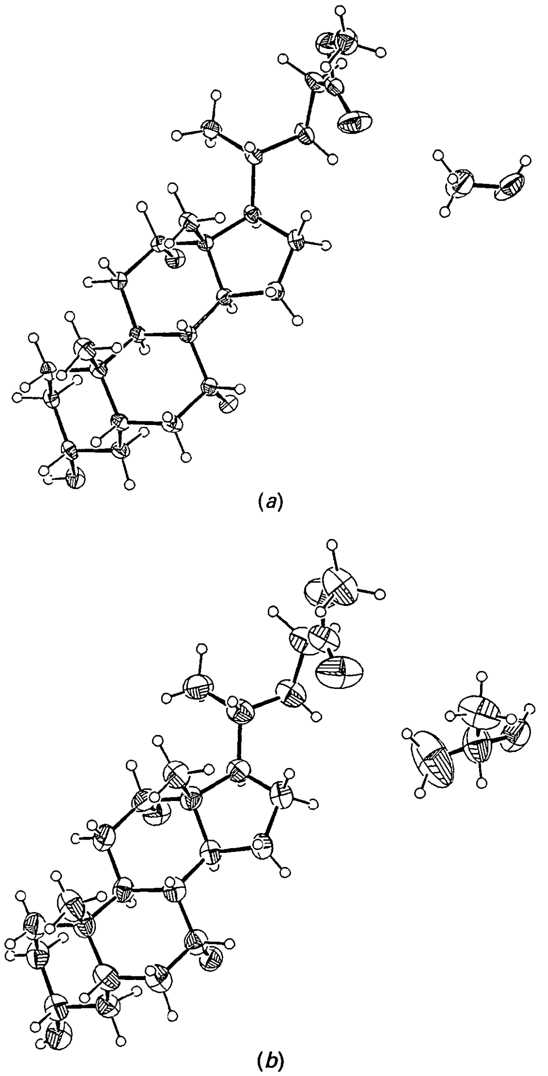


Fig. 1. *ORTEP* drawings (Johnson, 1976) of the molecular structures of MC and the alcohols. Non-H atoms are represented by thermal ellipsoids with 30% probability levels, whereas H atoms are drawn as spheres with $B = 1.0$ Å^2. (*a*) MC with methanol (1), (*b*) MC with 2-propanol (2).

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* Lists of anisotropic temperature factors for non-H atoms, atomic parameters for H atoms, structure factors, and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54554 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0515]

Table 2. *Bond distances* (Å) *and angles* (°) *for non-*H *atoms and hydrogen-bond parameters* (Å, °) *with e.s.d.'s in parentheses*

	(1)	(2)
C(1)—C(2)	1.533 (5)	1.531 (10)
C(1)—C(10)	1.542 (5)	1.550 (11)
C(2)—C(3)	1.510 (5)	1.496 (10)
C(3)—C(4)	1.506 (5)	1.515 (10)
C(3)—O(25)	1.436 (4)	1.444 (9)
C(4)—C(5)	1.543 (5)	1.533 (10)
C(5)—C(6)	1.537 (5)	1.530 (11)
C(5)—C(10)	1.546 (5)	1.552 (11)
C(6)—C(7)	1.529 (5)	1.529 (10)
C(7)—C(8)	1.527 (5)	1.531 (9)
C(7)—O(29)	1.447 (4)	1.431 (8)
C(8)—C(9)	1.546 (4)	1.560 (8)
C(8)—C(14)	1.527 (4)	1.516 (8)
C(9)—C(10)	1.563 (4)	1.557 (10)
C(9)—C(11)	1.546 (5)	1.524 (9)
C(10)—C(19)	1.549 (5)	1.551 (13)
C(11)—C(12)	1.537 (5)	1.545 (10)
C(12)—C(13)	1.537 (4)	1.538 (9)
C(12)—O(26)	1.448 (4)	1.434 (8)
C(13)—C(14)	1.540 (4)	1.526 (8)
C(13)—C(17)	1.555 (4)	1.556 (9)
C(13)—C(18)	1.541 (5)	1.545 (10)
C(14)—C(15)	1.518 (5)	1.543 (9)
C(15)—C(16)	1.542 (5)	1.545 (10)
C(16)—C(17)	1.557 (5)	1.544 (9)
C(17)—C(20)	1.550 (5)	1.549 (10)
C(20)—C(21)	1.527 (5)	1.496 (12)
C(20)—C(22)	1.538 (5)	1.532 (12)
C(22)—C(23)	1.532 (6)	1.530 (13)
C(23)—C(24)	1.494 (6)	1.505 (13)
C(24)—O(27)	1.194 (6)	1.182 (13)
C(24)—O(28)	1.337 (5)	1.285 (12)
O(28)—C(30)	1.442 (6)	1.448 (15)
C(2)—C(1)—C(10)	115.1 (3)	114.4 (6)
C(1)—C(2)—C(3)	109.1 (3)	111.2 (6)
C(2)—C(3)—C(4)	110.7 (3)	109.5 (6)
C(2)—C(3)—C(25)	112.6 (3)	110.8 (6)
C(4)—C(3)—C(25)	106.6 (3)	110.9 (6)
C(3)—C(4)—C(5)	112.4 (3)	112.0 (6)
C(4)—C(5)—C(6)	110.3 (3)	110.5 (6)
C(4)—C(5)—C(10)	113.5 (3)	114.0 (6)
C(6)—C(5)—C(10)	113.3 (3)	112.9 (6)
C(5)—C(6)—C(7)	114.6 (3)	113.7 (6)
C(6)—C(7)—C(8)	111.2 (3)	110.6 (6)
C(6)—C(7)—C(29)	111.5 (3)	110.7 (6)
C(8)—C(7)—C(29)	112.1 (3)	111.1 (5)
C(7)—C(8)—C(9)	111.4 (3)	110.2 (5)
C(7)—C(8)—C(14)	112.1 (3)	112.4 (5)
C(9)—C(8)—C(14)	111.7 (3)	110.8 (5)
C(8)—C(9)—C(10)	111.1 (3)	110.9 (5)
C(8)—C(9)—C(11)	112.4 (3)	112.4 (5)
C(10)—C(9)—C(11)	114.2 (3)	114.1 (6)
C(1)—C(10)—C(5)	108.7 (3)	108.8 (6)
C(1)—C(10)—C(9)	112.2 (3)	111.4 (6)
C(1)—C(10)—C(19)	106.4 (3)	107.4 (7)
C(5)—C(10)—C(9)	108.9 (3)	108.5 (6)
C(5)—C(10)—C(19)	109.4 (3)	109.0 (7)
C(9)—C(10)—C(19)	111.1 (3)	111.6 (7)
C(9)—C(11)—C(12)	115.0 (3)	115.2 (6)
C(11)—C(12)—C(13)	111.1 (3)	111.2 (5)
C(11)—C(12)—O(26)	110.1 (3)	111.3 (5)
C(13)—C(12)—O(26)	109.0 (3)	111.2 (5)
C(12)—C(13)—C(14)	107.0 (3)	107.5 (5)
C(12)—C(13)—C(17)	117.6 (3)	118.2 (5)
C(12)—C(13)—C(18)	109.4 (3)	108.8 (5)
C(14)—C(13)—C(17)	100.6 (3)	100.9 (5)
C(14)—C(13)—C(18)	112.2 (3)	112.3 (5)
C(17)—C(13)—C(18)	109.7 (3)	109.1 (5)
C(8)—C(14)—C(13)	113.7 (3)	115.7 (5)
C(8)—C(14)—C(15)	117.8 (3)	117.2 (5)
C(13)—C(14)—C(15)	104.2 (3)	104.4 (5)
C(14)—C(15)—C(16)	104.4 (3)	103.1 (5)
C(15)—C(16)—C(17)	106.7 (3)	107.9 (6)
C(13)—C(17)—C(16)	103.5 (3)	102.9 (5)
C(13)—C(17)—C(20)	118.3 (3)	119.5 (6)
C(16)—C(17)—C(20)	112.0 (3)	113.2 (6)
C(17)—C(20)—C(21)	112.3 (3)	114.1 (7)
C(17)—C(20)—C(22)	111.3 (3)	110.0 (6)
C(21)—C(20)—C(22)	109.5 (3)	110.6 (7)
C(20)—C(22)—C(23)	112.1 (3)	114.0 (7)
C(22)—C(23)—C(24)	112.6 (4)	114.2 (8)
C(23)—C(24)—O(27)	126.3 (4)	126.6 (9)
C(23)—C(24)—O(28)	111.7 (4)	111.1 (8)
O(27)—C(24)—O(28)	121.9 (4)	122.2 (9)
C(24)—O(28)—C(30)	117.1 (4)	115.8 (9)

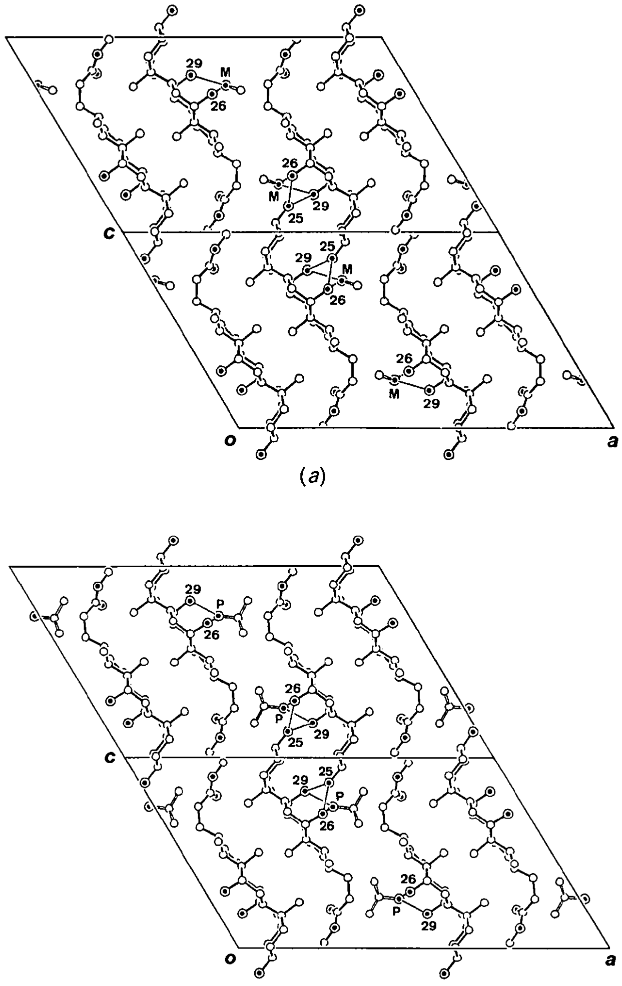
O—H···O		O···O	O—H···O
O(25)—H(25)^{i}···O(26)^{ii}	(1)	2.777 (4)	169 (5)
	(2)	2.817 (7)	165 (10)
O(26)—H(26)^{ii}···O(*M*/*P*)^{iii}	(1)	2.692 (5)	174 (5)
	(2)	2.682 (9)	176 (8)
O(*M*/*P*)—H(*M*/*P*)^{iii}···O(29)^{iv}	(1)	2.749 (5)	145 (6)
	(2)	2.727 (9)	162 (8)
O(29)—H(29)^{iv}···O(25)^{v}	(1)	2.847 (4)	165 (5)
	(2)	2.913 (7)	173 (7)

Symmetry code: (i) x, y, z; (ii) $-x, y, -z$; (iii) $x, -1+y, 1+z$; (iv) $-x, -1+y, -z$; (v) $x, -1+y, z$.

Discussion. The molecular structures of (1) and (2) drawn by *ORTEP* (Johnson, 1976) are presented in Fig. 1. The molecular conformations of MC are quite similar to each other. But, the conformations of the carboxy ester moiety [the so-called 'tail' part described by Miki *et al.* (1988)] are different from those found in the crystal structures of CA and MDC; the C(20)—C(22)—C(23)—C(24) torsion angles in the MC compounds are 78.5 (4)° for (1) and 74.5 (10)° for (2), while they are 171.8 (4)° for CA (Miki, Kasai, Shibakami, Chirachanchai, Takemoto & Miyata, 1990) and 179.8 (10) and 163.7 (7)° for MDC (Miki *et al.*, 1989). The conformation of this tail part is very important with regard to inclusion phenomena (Miki *et al.*, 1991). Bond distances and angles, listed in Table 2, show normal values for steroidal compounds within experimental errors.

The crystal structures are presented in Fig. 2. The arrangement of the MC molecules is similar in both compounds. The guest alcohol molecules, which are hydrogen bonded to the host MC molecules, are located at essentially the same positions in both crystal structures. The MC molecules form anti-parallel layers along the direction of the crystallographic c axis. Such an anti-parallel layered structure has been found in crystals of the inclusion compound of CA (Miki *et al.*, 1988), while a parallel arrangement of the layers was observed in the case of DCA (Giglio, 1984). However, any channel spaces for guest molecules which were found in many of the inclusion compounds of both DCA and CA were not formed by the host MC molecules in the present crystal structures. This molecular arrangement of MC is also different from that of MDC found in the addition compound with methanol (Miki *et al.*, 1989).

In both crystals (1) and (2), four kinds of hydrogen bonds are formed not only between MC molecules but also between MC and alcohol molecules, providing a network with the sequence —O(25)—H(x, y, z)···O(26)—H($-x$, y, $-z$)···O(M/P)—H(x, $-1+y$, $1+z$)···O(29)—H($-x$, $-1+y$, $-z$)···O(25)—H(x, $-1+y$, z). Among the four O atoms that can contribute as hydrogen donors and acceptors, only O(27) is not included in the present hydrogen-bond network. The thermal motion of the O(27) atom in each crystal is extremely high compared with those of other O atoms. Such an observation for the non-hydrogen-bonded O atom was also found in the crystal of MDC with methanol (Miki *et al.*, 1989). The parameters of this hydrogen-bond network are also listed in Table 2.



(*a*)

(*b*)

Fig. 2. *ORTEP* drawings (Johnson, 1976) of the crystal structures of 1:1 addition compounds between MC and the alcohols as viewed along the *b* axis. C and O atoms are represented by empty and half-filled circles, respectively. H atoms are omitted for clarity. The hydrogen-bond network is shown by thin lines together with the numbering of the O atoms involved in the network, where dotted lines between O(M/P) and O(26) show hydrogen bonds with upper or lower molecules in the direction of the *b* axis. (*a*) MC with methanol (1), (*b*) MC with 2-propanol (2).

It can be concluded that the introduction of the methyl group at O(28) of CA caused a new type of the arrangement of the host steroid molecules. The crystal structures of CA with methanol and 2-propanol display different types of packing (orthorhombic, space group $P2_12_12_1$) (Johnson & Schaefer, 1972; Jones & Nassimbeni, 1990). Esterification of CA introduces a comparatively bulky methyl group and eliminates the hydrogen which is capable of acting as a donor in hydrogen bonds. This is an important factor which significantly changes the molecular arrangement of the host bile acids.

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References

Ashida, T. (1979). *HBLS-V. The Universal Crystallographic Computing System–Osaka*, pp. 53–59. Computation Center, Osaka Univ., Japan.

Giglio, E. (1984). *Inclusion Compounds*, Vol. II, edited by J. L. Atwood, J. E. D. Davies & D. D. MacNicol, pp. 207–229. London: Academic Press.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Johnson, P. L. & Schaefer, J. P. (1972). *Acta Cryst.* **B28**, 3083–3086.

Jones, E. L. & Nassimbeni, L. R. (1990). *Acta Cryst.* **B46**, 399–405.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Miki, K., Kasai, N., Shibakami, M., Chirachanchai, S., Takemoto, K. & Miyata, M. (1990). *Acta Cryst.* **C46**, 2442–2445.

Miki, K., Kasai, N., Shibakami, M., Takemoto, K. & Miyata, M. (1991). *J. Chem. Soc. Chem. Commun.* In the press.

Miki, K., Kasai, N., Tsutsumi, H., Miyata, M. & Takemoto, K. (1987). *J. Chem. Soc. Chem. Commun.* pp. 545–546.

Miki, K., Masui, A., Kasai, N., Miyata, M., Goonewardena, W., Shibakami, M. & Takemoto, K. (1989). *Acta Cryst.* **C45**, 79–83.

Miki, K., Masui, A., Kasai, N., Miyata, M., Shibakami, M. & Takemoto, K. (1988). *J. Am. Chem. Soc.* **110**, 6594–6596.

Miyata, M., Goonewardena, W., Shibakami, M., Takemoto, K., Masui, A., Miki, K. & Kasai, N. (1987). *J. Chem. Soc. Chem. Commun.* pp. 1140–1141.

Miyata, M., Shibakami, M., Chirachanchai, S., Takemoto, K., Kasai, N. & Miki, K. (1990). *Nature (London)*, **343**, 446–447.