Table 3. *Bond angles* (°) *involving the non*-H *atoms with e.s.d.'s in parentheses*

Br(3)–Br(2)–C(3)	38·9 (2)	Br(2)–Br(3)–C(3)	39·0 (2)
C(2)–C(1)–C(8)	109·4 (7)	C(2)–C(1)–C(7)	112·6 (7)
C(8)–C(1)–C(7)	100·7 (8)	Br(1)–C(2)–C(1)	107·5 (6)
Br(1)–C(2)–C(3)	112·8 (5)	C(1)–C(2)–C(3)	112·5 (8)
Br(2)–C(3)–Br(3)	102·1 (4)	Br(2)–C(3)–C(2)	113·7 (6)
Br(3)–C(3)–C(2)	107·2 (5)	Br(2)–C(3)–C(4)	106·9 (5)
Br(3)–C(3)–C(4)	110·6 (6)	C(2)–C(3)–C(4)	115·5 (7)
C(3)–C(4)–C(5)	112·6 (7)	C(4)–C(5)–C(8)	109·1 (8)
C(4)–C(5)–C(6)	112·6 (7)	C(8)–C(5)–C(6)	100·5 (7)
C(1)–C(8)–C(5)	99·3 (7)	C(1)–C(7)–C(6)	107·7 (7)
C(1)–C(7)–C(12)	132·1 (9)	C(6)–C(7)–C(12)	120·2 (8)
C(5)–C(6)–C(7)	108·4 (8)	C(5)–C(6)–C(9)	132·3 (9)
C(7)–C(6)–C(9)	119·2 (8)	C(6)–C(9)–C(10)	118·7 (11)
C(9)–C(10)–C(11)	122·9 (11)	C(10)–C(11)–C(12)	119·3 (9)
C(7)–C(12)–C(11)	119·6 (9)		

shorter than 1·39 Å, the characteristic bond length of a C–C bond in an aromatic molecule. The other bonds of the benzene ring are nearly 1·39 Å. All of the other C–C single-bond lengths of the molecule range from 1·49 to 1·55 Å with a mean value of 1·53 Å. The three C–Br bonds are very nearly equal (1·98 Å) and agree with those observed in dibromocyclopertilide (Nagumo, Kawai & Iitaka, 1982). In the refinement, we could not find the coordinates of the two H atoms of both C(4) and C(8) from the difference Fourier maps. This is probably due to the motion of these H atoms. So, applying an interatomic distance constraint of 1·01782 Å with an e.s.d. of 0·07 Å to the C–H bond, the positions and isotropic temperature factors of these H atoms were refined.

The addition of bromine to unsaturated bicyclic systems can lead to a multiplicity of products. This kind of system can easily undergo Wagner–Meerwein rearrangement to give rearranged and unrearranged products (Provolotskaya, Limasova, Berus, Exner & Barkash, 1969). X-ray structural determination of (2) has confirmed that one part of this bromination at 273 K proceeds without rearrangement. Furthermore, it was found that the bromine of C(2) is in the *exo* position. From the *exo* configuration of this bromine, we are not able to predict whether the initial attack of bromine on the double bond in (2) is *endo* or *exo*.

The authors thank the Scientific and Technical Research Council of Turkey for financial support, Institut für Kristallographie der Universität Karlsruhe for the use of the diffractometer and Mr G. Mattern for his skilled technical assistance.

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Structure of a 2:1 Addition Compound of Methyl Deoxycholate with Methanol

BY KUNIO MIKI,* AKIHIKO MASUI AND NOBUTAMI KASAI*

Department of Applied Chemistry, Faculty of Engineering, Osaka University, Yamadaoka, Suita, Osaka 565, *Japan*

AND MIKIJI MIYATA, WYJAYANTHI GOONEWARDENA, MOTONARI SHIBAKAMI AND KIICHI TAKEMOTO

Department of Applied Fine Chemistry, Faculty of Engineering, Osaka University, Yamadaoka, Suita, Osaka 565, *Japan*

(*Received* 31 *May* 1988; *accepted* 1 *August* 1988)

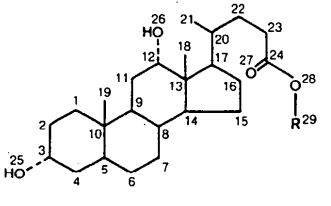
Abstract. Methyl 3α,12α-dihydroxy-5β-cholan-24-oate–methanol (2/1), $C_{25}H_{42}O_4.\frac{1}{2}CH_4O$, $M_r = 422{\cdot}63$, monoclinic, $P2_1$, $a = 16{\cdot}543$ (3), $b = 12{\cdot}109$ (3), $c = 12{\cdot}477$ (1) Å, $\beta = 102{\cdot}12$ (1)°, $U = 2443{\cdot}6$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}149$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}13$ cm^{-1}, $F(000) = 932$, $T = 295$ K, $R = 0{\cdot}062$ for 3135 observed reflections. The molar ratio between methyl deoxycholate (MDC) and methanol is 2:1. The

* To whom correspondence should be addressed.

0108-2701/89/010079-05$03.00

layered arrangement observed previously in the inclusion compounds of deoxycholic acid, in which hydrophobic channels are provided by the steroidal host molecules, is not observed in the present MDC crystal structure. The hydrogen-bond network is rigidly formed not only between MDC molecules but also with the methanol molecules.

Introduction. Among lattice-type inclusion compounds, it is well known that the steroidal bile acid, deoxycholic acid (DCA), provides tunnel-like spaces for guest molecules usually called 'channels' in which a wide variety of organic molecules can be accommodated (Giglio, 1984). Recently, we have found that these DCA channels can accept not only organic molecules but also an organometallic compound such as ferrocene (Miki, Kasai, Tsutsumi, Miyata & Takemoto, 1987). However, little is known about the inclusion ability of other steroidal bile acids (Herndon, 1967). A series of addition compounds of several bile acids and their derivatives with various organic guest molecules has been investigated in order to develop new types of host molecules for inclusion compounds (Miyata, Shibakami, Goonewardena & Takemoto, 1987; Miyata, Goonewardena, Shibakami, Takemoto, Masui, Miki & Kasai, 1987; Miki, Masui, Kasai, Miyata, Shibakami & Takemoto, 1988). Here we report the crystal structure of an addition compound of the methyl ester of DCA, methyl deoxycholate (MDC), with methanol obtained by cocrystallization.



Deoxycholic acid (DCA) $R = H$
Methyl deoxycholate (MDC) $R = CH_3$

Experimental. Crystals of the title compound were grown as colorless prisms from a methanol solution of MDC. A well shaped crystal with approximate dimensions of 0·25 × 0·25 × 0·30 mm was mounted on a Rigaku automated four-circle diffractometer and studied with Ni-filtered Cu $K\alpha$ radiation (λ = 1·5418 Å). Accurate unit-cell parameters were determined by a least-squares fit of 2θ values of 23 centered reflections in the 2θ range 38·8–77·5°. Intensities were measured by the θ–2θ scan technique with a scan rate of 4° min^{-1} in 2θ and a scan width of $\Delta(2\theta) = (1{\cdot}6 + 0{\cdot}28\tan\theta)°$. Background intensities were measured for 5 s at each end of a scan. Three standard reflections (040, 600, 006) were remeasured after every 61 reflections; no significant loss of intensity was observed throughout data collection. 3600 independent reflections ($R_{int} = 0{\cdot}014$) were collected with 2θ up to 116·0° ($\sin\theta/\lambda = 0{\cdot}550$ Å^{-1}) and index range of h −18 to 17, k 0 to 13, l 0 to 13. Corrections for Lorentz and polarization effects were applied to the intensity data; no absorption or extinction corrections were carried out.

The structure was solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The initial E map gave partial structures of two crystallographically independent MDC molecules, that is, for 28 of the 58 non-H atoms. Positions of the remaining non-H atoms of MDC were located stepwise from the subsequent Fourier syntheses. Two non-H atoms of methanol were also found from the Fourier map based on all the non-H-atom positions of MDC. This indicated that the molar ratio between MDC and methanol is 2 : 1.

The structure was refined by the block-diagonal least-squares procedure using the *HBLS*V program (Ashida, 1979). 3135 observed reflections [$|F_o| \geq 3\sigma(|F_o|)$] were included in the refinement; the function minimized was $\sum w(|F_o| - |F_c|)^2$. On the difference Fourier map calculated after several cycles of anisotropic refinement, positions of all the H atoms were reasonably found at essentially the same positions as those estimated on the basis of stereochemical considerations. These H-atom parameters were included to calculate structure factors in the further refinement cycles. The weighting scheme used was $w = [\sigma(F_o)^2 + 0{\cdot}1030|F_o|]^{-1}$, although unit weights were employed in the early stage of the refinements. The number of observations per refined parameter is 3135/542 = 5·78 and $S = 0{\cdot}88$. The final R and wR values are 0·062 and 0·085, respectively. $(\Delta/\sigma)_{max}$ in the final refinement cycle was 0·10 and 0·39 for positional and thermal parameters, respectively. The peaks in the final $\Delta\rho$ map were between 0·4 and −0·1 e Å^{-3}. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are listed in Table 1.* The O(27) atom of molecule A showed an abnormally large thermal motion. Although we examined the possibility of disorder in this ester chain, no significant peaks were found in the difference Fourier map (see also *Discussion*).

All the computations were performed on an ACOS 850 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

Discussion. The molecular structures of two crystallographically independent MDC molecules drawn by

*Lists of anisotropic temperature factors for non-H atoms, atomic parameters for H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51303 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*
(*a*) MDC molecule *A*				
C(1)	0·4716 (4)	1·0746 (6)	0·6720 (6)	4·8
C(2)	0·5041 (4)	1·0427 (6)	0·7897 (5)	4·5
C(3)	0·5702 (4)	0·9542 (6)	0·7940 (6)	5·1
C(4)	0·5325 (4)	0·8539 (6)	0·7311 (6)	5·1
C(5)	0·4919 (4)	0·8826 (7)	0·6125 (5)	4·9
C(6)	0·4547 (5)	0·7801 (7)	0·5483 (6)	5·9
C(7)	0·3729 (4)	0·7444 (6)	0·5793 (6)	5·0
C(8)	0·3109 (4)	0·8383 (6)	0·5633 (5)	4·2
C(9)	0·3484 (4)	0·9404 (5)	0·6310 (5)	3·9
C(10)	0·4294 (4)	0·9791 (6)	0·6000 (5)	4·6
C(11)	0·2842 (4)	1·0314 (5)	0·6264 (5)	4·5
C(12)	0·2013 (4)	0·9963 (5)	0·6526 (5)	4·1
C(13)	0·1655 (4)	0·8977 (6)	0·5798 (5)	4·2
C(14)	0·2304 (4)	0·8070 (5)	0·5963 (5)	4·2
C(15)	0·1839 (5)	0·7039 (6)	0·5403 (6)	5·6
C(16)	0·0949 (5)	0·7199 (7)	0·5567 (7)	6·2
C(17)	0·0922 (4)	0·8352 (6)	0·6118 (5)	4·7
C(18)	0·1401 (5)	0·9383 (7)	0·4603 (5)	5·6
C(19)	0·4128 (5)	1·0201 (7)	0·4804 (6)	6·0
C(20)	0·0038 (4)	0·8831 (8)	0·5873 (6)	6·3
C(21)	0·0022 (5)	1·0004 (7)	0·6380 (7)	6·5
C(22)	−0·0562 (5)	0·8049 (8)	0·6391 (8)	7·3
C(23)	−0·1441 (6)	0·8322 (12)	0·6034 (10)	9·8
C(24)	−0·1986 (5)	0·7598 (10)	0·6519 (9)	9·3
O(25)	0·6067 (3)	0·9219 (5)	0·9052 (4)	6·4
O(26)	0·2136 (3)	0·9623 (4)	0·7660 (3)	4·8
O(27)	−0·2651 (8)	0·7741 (20)	0·6335 (16)	30·9
O(28)	−0·1705 (4)	0·6953 (8)	0·7321 (7)	10·5
C(29)	−0·2284 (6)	0·6287 (10)	0·7748 (8)	8·5
(*b*) MDC molecule *B*				
C(1)	0·0937 (5)	0·3983 (7)	0·7646 (5)	5·6
C(2)	0·1565 (5)	0·3045 (7)	0·7952 (6)	6·0
C(3)	0·1256 (5)	0·2220 (7)	0·8687 (6)	5·9
C(4)	0·1057 (5)	0·2761 (7)	0·9686 (6)	5·7
C(5)	0·0450 (4)	0·3723 (7)	0·9398 (6)	5·2
C(6)	0·0262 (4)	0·4244 (7)	1·0446 (6)	5·5
C(7)	0·1001 (4)	0·4913 (7)	1·1066 (5)	5·0
C(8)	0·1302 (3)	0·5760 (6)	1·0352 (5)	4·0
C(9)	0·1490 (3)	0·5249 (5)	0·9307 (4)	3·6
C(10)	0·0731 (4)	0·4588 (6)	0·8645 (5)	4·6
C(11)	0·1840 (4)	0·6097 (6)	0·8620 (5)	4·6
C(12)	0·2607 (4)	0·6720 (6)	0·9254 (5)	4·2
C(13)	0·2421 (4)	0·7247 (5)	1·0304 (5)	3·7
C(14)	0·2093 (4)	0·6346 (5)	1·0967 (4)	3·5
C(15)	0·2088 (4)	0·6888 (6)	1·2073 (5)	5·0
C(16)	0·2862 (4)	0·7661 (7)	1·2254 (5)	4·9
C(17)	0·3189 (4)	0·7635 (6)	1·1178 (5)	3·8
C(18)	0·1816 (4)	0·8213 (6)	0·9973 (6)	4·8
C(19)	0·0007 (4)	0·5389 (8)	0·8175 (6)	6·0
C(20)	0·3653 (4)	0·8712 (6)	1·0994 (5)	4·6
C(21)	0·3868 (5)	0·8742 (8)	0·9874 (6)	6·4
C(22)	0·4453 (4)	0·8753 (7)	1·1894 (5)	5·2
C(23)	0·4924 (5)	0·9833 (7)	1·1963 (7)	6·9
C(24)	0·5799 (4)	0·9717 (7)	1·2613 (6)	5·5
O(25)	0·1837 (4)	0·1331 (5)	0·9010 (5)	7·7
O(26)	0·3292 (2)	0·5974 (4)	0·9550 (3)	4·6
O(27)	0·6384 (4)	1·0059 (7)	1·2357 (5)	8·8
O(28)	0·5829 (3)	0·9207 (7)	1·3535 (5)	8·8
C(29)	0·6652 (6)	0·8989 (12)	1·4193 (9)	10·0
(*c*) Methanol				
C(*M*)	0·3857 (7)	0·2064 (11)	0·9460 (10)	10·2
O(*M*)	0·3336 (4)	0·2224 (6)	1·0145 (5)	7·7

* As defined by Hamilton (1959).

*ORTEP*II (Johnson, 1976) are presented in Fig. 1. Molecules *A* and *B* have an essentially similar structure except for slight conformational changes in the methylene and ester chains and in the H atoms of the OH groups. Bond distances and angles are presented in Table 2, all of which show the normal values for the standard steroidal compounds within experimental errors.

The crystal packing diagram is presented in Fig. 2. In several crystal structures of DCA so far determined in which the channel spaces for guest molecules are provided, the DCA molecules are arranged in a one-dimensional array so that the OH and CH_3 groups gather together to form hydrophilic and hydrophobic layers, respectively. In the present crystal structure of MDC, similar layered arrangements are partially observed along the *a* axis only near molecules *A*, where the methyl groups attached to the steroidal skeleton of MDC are associated with each other to provide the hydrophobic groove. Molecules *B* do not obey such layered arrangements. In addition, channel spaces for the guest molecules are not provided. The methanol molecules contact with both molecules *A* and *B* by hydrogen bonding.

In the case of inclusion compounds of DCA, four hydrogen bonds are formed between the host molecules to give stable and rigid hydrophobic channel walls (Giglio, 1984). In the MDC structure, as shown in Fig. 2, five kinds of hydrogen bonds link not only the MDC host molecules but also the methanol molecules, providing a network with the sequence —O(26*A*)—

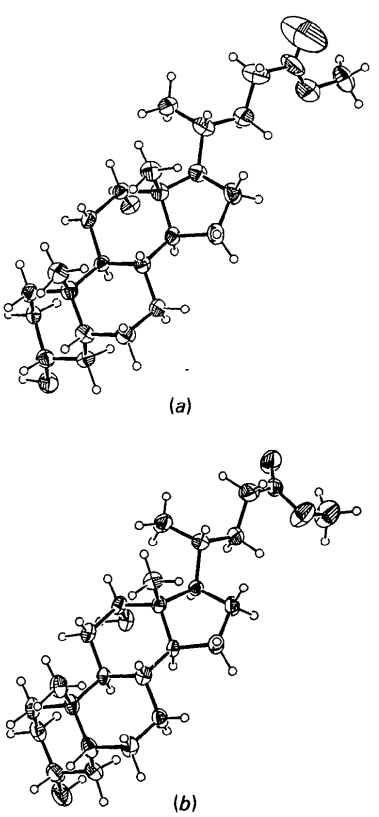


Fig. 1. *ORTEP* (Johnson, 1976) drawing of the molecular structures of the two crystallographically independent MDC molecules. Non-H atoms are represented by thermal ellipsoids with 30% probability levels, whereas H atoms are drawn by a sphere with $B = 1{\cdot}0$ Å^2. (*a*) Molecule *A*, (*b*) molecule *B*.

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

	MDC molecule *A*	MDC molecule *B*
C(1)–C(2)	1·505 (9)	1·533 (12)
C(1)–C(10)	1·538 (10)	1·545 (11)
C(2)–C(3)	1·523 (10)	1·515 (11)
C(3)–C(4)	1·508 (10)	1·504 (11)
C(3)–O(25)	1·447 (9)	1·443 (10)
C(4)–C(5)	1·531 (11)	1·530 (11)
C(5)–C(6)	1·534 (11)	1·541 (12)
C(5)–C(10)	1·545 (10)	1·542 (11)
C(6)–C(7)	1·545 (11)	1·533 (12)
C(7)–C(8)	1·517 (10)	1·510 (10)
C(8)–C(9)	1·551 (9)	1·532 (9)
C(8)–C(14)	1·521 (9)	1·544 (9)
C(9)–C(10)	1·545 (9)	1·570 (10)
C(9)–C(11)	1·522 (9)	1·528 (10)
C(10)–C(19)	1·541 (11)	1·557 (12)
C(11)–C(12)	1·536 (9)	1·543 (10)
C(12)–C(13)	1·541 (9)	1·545 (9)
C(12)–O(26)	1·446 (8)	1·436 (9)
C(13)–C(14)	1·520 (9)	1·535 (9)
C(13)–C(17)	1·551 (10)	1·563 (9)
C(13)–C(18)	1·542 (11)	1·538 (9)
C(14)–C(15)	1·554 (10)	1·530 (10)
C(15)–C(16)	1·540 (11)	1·564 (11)
C(16)–C(17)	1·561 (11)	1·549 (10)
C(17)–C(20)	1·544 (12)	1·555 (10)
C(20)–C(21)	1·557 (13)	1·512 (12)
C(20)–C(22)	1·604 (14)	1·546 (10)
C(22)–C(23)	1·466 (17)	1·515 (12)
C(23)–C(24)	1·475 (19)	1·510 (12)
C(24)–O(27)	1·091 (27)	1·157 (12)
C(24)–O(28)	1·277 (15)	1·297 (12)
O(28)–C(29)	1·437 (15)	1·459 (17)
	Methanol	
C(*M*)–O(*M*)	1·350 (15)	

	MDC molecule *A*	MDC molecule *B*
C(2)–C(1)–C(10)	113·8 (6)	113·8 (7)
C(1)–C(2)–C(3)	108·7 (6)	110·0 (7)
C(2)–C(3)–C(4)	109·5 (6)	112·1 (7)
C(2)–C(3)–O(25)	112·1 (6)	111·7 (7)
C(4)–C(3)–O(25)	108·9 (6)	110·1 (7)
C(3)–C(4)–C(5)	111·6 (6)	112·6 (7)
C(4)–C(5)–C(6)	111·5 (7)	110·6 (7)
C(4)–C(5)–C(10)	114·7 (6)	113·2 (7)
C(6)–C(5)–C(10)	111·9 (6)	112·3 (7)
C(5)–C(6)–C(7)	111·8 (7)	111·2 (7)
C(6)–C(7)–C(8)	111·1 (6)	112·8 (7)
C(7)–C(8)–C(9)	110·1 (6)	112·2 (6)
C(7)–C(8)–C(14)	112·6 (6)	111·7 (6)
C(9)–C(8)–C(14)	108·9 (5)	107·8 (5)
C(8)–C(9)–C(10)	112·0 (5)	111·8 (5)
C(8)–C(9)–C(11)	111·1 (5)	112·0 (6)
C(10)–C(9)–C(11)	114·0 (5)	113·7 (6)
C(1)–C(10)–C(5)	107·3 (6)	108·4 (6)
C(1)–C(10)–C(9)	113·1 (6)	112·3 (6)
C(1)–C(10)–C(19)	106·9 (6)	105·9 (6)
C(5)–C(10)–C(9)	110·0 (6)	109·6 (6)
C(5)–C(10)–C(19)	108·8 (6)	110·3 (6)
C(9)–C(10)–C(19)	110·6 (6)	110·3 (6)
C(9)–C(11)–C(12)	116·0 (5)	114·1 (6)
C(11)–C(12)–C(13)	109·7 (5)	110·4 (6)
C(11)–C(12)–O(26)	109·8 (5)	110·1 (6)
C(13)–C(12)–O(26)	108·3 (5)	109·2 (6)
C(12)–C(13)–C(14)	107·9 (6)	108·6 (5)
C(12)–C(13)–C(17)	116·7 (6)	116·0 (5)
C(12)–C(13)–C(18)	108·4 (6)	108·7 (6)
C(14)–C(13)–C(17)	100·4 (6)	99·5 (5)
C(14)–C(13)–C(18)	113·8 (6)	113·7 (5)
C(17)–C(13)–C(18)	109·6 (6)	110·3 (5)
C(8)–C(14)–C(13)	114·6 (6)	114·7 (5)
C(8)–C(14)–C(15)	117·3 (6)	118·7 (6)
C(13)–C(14)–C(15)	104·5 (6)	104·5 (5)
C(14)–C(15)–C(16)	103·7 (6)	103·2 (6)
C(15)–C(16)–C(17)	106·7 (7)	106·7 (6)
C(13)–C(17)–C(16)	103·1 (6)	103·3 (5)
C(13)–C(17)–C(20)	121·7 (7)	120·2 (6)
C(16)–C(17)–C(20)	111·2 (7)	112·6 (6)
C(17)–C(20)–C(21)	111·0 (7)	112·1 (6)
C(17)–C(20)–C(22)	109·8 (7)	107·1 (6)

Table 2 (*cont.*)

	MDC molecule *A*	MDC molecule *B*
C(21)–C(20)–C(22)	108·1 (8)	109·8 (6)
C(20)–C(22)–C(23)	113·7 (9)	114·9 (7)
C(22)–C(23)–C(24)	113·2 (11)	112·0 (7)
C(23)–C(24)–O(27)	119·7 (17)	125·5 (8)
C(23)–C(24)–O(28)	122·1 (11)	112·0 (8)
O(27)–C(24)–O(28)	116·6 (17)	122·4 (9)
C(24)–O(28)–C(29)	118·1 (10)	116·3 (9)

O–H···O	O···O	O–H	H···O	O–H···O
O(26*A*^{ii})–H(26*A*^{ii})···O(25*B*^{i})	2·777 (8)	1·00	1·78	174
O(25*B*^{i})–H(25*B*^{i})···O(*M*^{i})	2·801 (9)	1·02	1·79	176
O(*M*^{i})–H(MD^{i})···O(25*A*^{iii})	2·721 (9)	1·00	1·75	162
O(25*A*^{iii})–H(25*A*^{iii})···O(26*B*^{i})	2·812 (8)	1·01	1·81	171
O(26*B*^{i})–H(26*B*^{i})···O(27*B*^{iii})	2·778 (10)	1·00	1·78	177

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

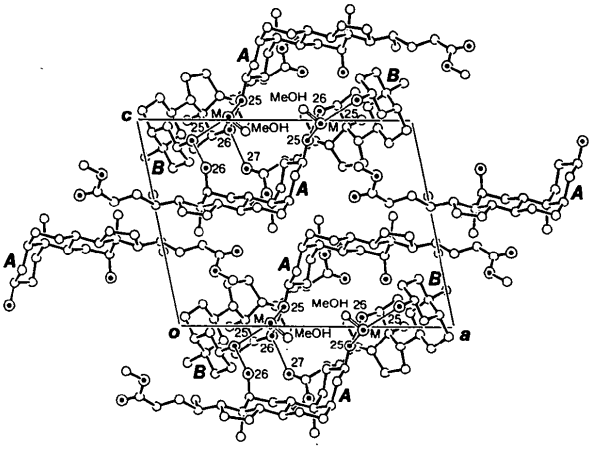


Fig. 2. Crystal structure of the addition compound between MDC and methanol as viewed along the *b* axis. C and O atoms are represented by empty and half-filled circles, respectively. H atoms are omitted for clarity. The two crystallographically independent MDC molecules are classified as *A* and *B*. The hydrogen-bond network is shown by thin lines, together with the numbering of the O atoms involved in hydrogen bonding.

H(26*A*)···O(25*B*)–H(25*B*)···O(*M*)–H(MD)···O(25*A*)–H(25*A*)···O(26*B*)–H(26*B*)···O(27*B*)=C(24*B*)–. The parameters for this hydrogen-bond network are listed in Table 2. Among the six O atoms that can contribute to hydrogen bonds, the O(27) atom of molecule *A*, which has high thermal motion, is the only O atom excluded from the hydrogen-bonding network.

It can be concluded that the esterification by the methyl group on the O(28) position of DCA induces a significant change in the crystal structure. The hydrophobic channel space for the methanol molecules is no longer provided in MDC. The O(28) positions take part in the edge of the channel wall in the crystal structures of DCA. In the MDC crystal structure, no channel walls can be constructed with any reasonable packing forms owing to the bulkiness of the methyl group. The change induced in the packing diagram is large enough

to affect the hydrogen-bond network between the host molecules.

Part of this work was supported by the Grant-in-Aids for Scientific Research from the Ministry of Education, Science and Culture, Japan.

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α-*N*-*tert*-Butyloxycarbonyl-L-amino-(*N*-methyl)succinimide (Boc-L-Asu-NMe)

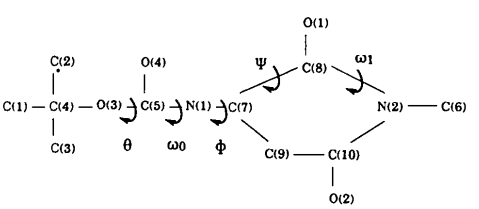
BY SANTE CAPASSO, MARIA P. NIOLA, FILOMENA SICA AND ADRIANA ZAGARI

Dipartimento di Chimica, Università di Napoli, Via Mezzocannone 4, 80134 *Napoli, Italy*

(*Received* 29 *March* 1988; *accepted* 25 *July* 1988)

Abstract. $C_{10}H_{16}N_2O_4$, $M_r = 228{\cdot}25$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}768$ (1), $b = 13{\cdot}428$ (1), $c = 16{\cdot}173$ (2) Å, $V = 1252{\cdot}6$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}21$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418, $\mu = 0{\cdot}75$ mm^{-1}, $F(000) = 488$, room temperature, final $R = 0{\cdot}061$ for 1126 observed reflections. The title compound is a model for the aminosuccinyl residue (Asu) in a peptide sequence. In the solid state it adopts a conformation similar to that found in Asu peptides folded in a type II′ β-bend structure. The succinimide ring deviates considerably from planarity and assumes an envelope conformation. In the crystal, chains of N—H···O hydrogen-bonded molecules wind up around the screw axes parallel to **a**.

Introduction. The crystal structure determination of the title compound is part of a research program on the conformational properties of peptides containing the succinimide ring, hereafter referred to as aminosuccinyl peptides or Asu peptides (Capasso, Mattia, Mazzarella & Zagari, 1984*a*,*b*; Capasso, Mazzarella, Sica & Zagari, 1984, 1987; Mazzarella, Schon, Sica & Zagari, 1988). A growing body of experimental data indicates that these peptides strongly prefer a folded conformation of the type II′ β-bend, stabilized by a 4→1 intramolecular hydrogen bond, with the Asu residue in the second position of the bend (Venkatachalam, 1968). As the characterizing fragment of these peptides is the Asu moiety, we have synthesized and studied by X-ray analysis the fully protected Boc-L-Asu-NMe molecule.



Experimental. Synthesis according to a procedure described elsewhere (Capasso, Mazzarella, Sica & Zagari, 1988). Crystal from ethyl acetate, 0·72 × 0·13 × 0·13 mm, Enraf–Nonius CAD-4F diffractometer, Ni-filtered radiation, lattice parameters from 25 reflections ($16 \le \theta \le 25°$); data collection $\omega/2\theta$ scan as suggested by peak-shape analysis, two intensity monitoring reflections (3% variation); 1418 independent reflections with $\theta < 70°$, $0 \le h \le 7$, $0 \le k \le 16$, $0 \le l \le 19$, 1126 with $I > 2{\cdot}5\sigma(I)$, Lp correction, absorption ignored. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), anisotropic full matrix (on F), H atoms from geometrical considerations, isotropic with the same B_{eq} as the atoms to which they

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