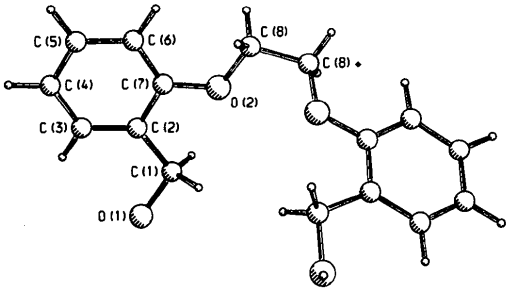

Fig. 1. View of the title compound.

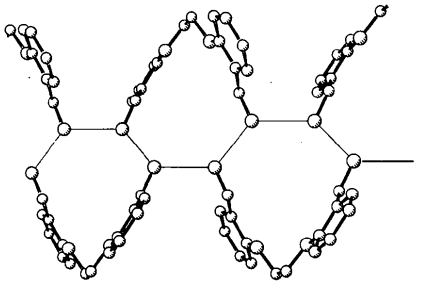

Fig. 2. Hydrogen-bond network across inversion centres.

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(1)	1·422 (3)	O(2)—C(7)	1·372 (3)
O(2)—C(8)	1·423 (3)	C(1)—C(2)	1·505 (4)
C(2)—C(3)	1·381 (4)	C(2)—C(7)	1·402 (4)
C(3)—C(4)	1·382 (4)	C(4)—C(5)	1·374 (5)
C(5)—C(6)	1·394 (4)	C(6)—C(7)	1·381 (4)
C(8)—C(8^{i})	1·489 (5)	O(1)···O(1^{ii})	2·731 (4)
O(1)···O(1^{iii})	2·741 (4)		

C(7)—O(2)—C(8)	118·7 (2)	O(1)—C(1)—C(2)	112·7 (2)
C(1)—C(2)—C(3)	123·7 (2)	C(1)—C(2)—C(7)	117·7 (2)
C(3)—C(2)—C(7)	118·6 (2)	C(2)—C(3)—C(4)	121·0 (3)
C(3)—C(4)—C(5)	119·7 (3)	C(4)—C(5)—C(6)	121·0 (3)
C(5)—C(6)—C(7)	118·6 (3)	O(2)—C(7)—C(2)	114·1 (2)
O(2)—C(7)—C(6)	124·7 (2)	C(2)—C(7)—C(6)	121·1 (2)
O(2)—C(8)—C(8^{i})	106·5 (2)	O(1^{ii})··O(1)··O(1^{iii})	128·4 (3)

Symmetry operations: (i) $1-x$, y, $-0{\cdot}5-z$; (ii) $1-x$, y, $0{\cdot}5-z$; (iii) $1-x$, $1-y$, $-z$.

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Structural Studies of Mitomycins. II. Structure of Mitomycin A Hemihydrate

BY NORIAKI HIRAYAMA* AND KUNIKATSU SHIRAHATA

Tokyo Research Laboratories, Kyowa Hakko Kogyo Co. Ltd, 3-6-6 *Asahimachi, Machida, Tokyo* 194, *Japan*

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Abstract. $C_{16}H_{20}N_3O_6.\frac{1}{2}H_2O$, $M_r = 359{\cdot}36$, monoclinic, $P2_1$, $a = 17{\cdot}893$ (2), $b = 10{\cdot}358$ (3), $c = 9{\cdot}029$ (1) Å, $\beta = 95{\cdot}16$ (1)°, $V = 1666{\cdot}6$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}40$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 8{\cdot}7$ cm^{-1}, $F(000) = 390$, $T = 293$ K, $wR = 0{\cdot}069$ for 3143 observed reflections with $F > 3\sigma(F)$. The benzoquinone ring deviates significantly from planarity. Its two O atoms are located on opposite sides of the least-squares plane through the benzene ring. Conformational differences between the two crystallographically independent molecules are observed around the substituent groups at C(7), C(9) and C(9*a*).

Introduction. Mitomycins are very effective antitumor antibiotics. To design more effective and less toxic mitomycins it is desirable to investigate the three-dimensional structures of mitomycins in detail. Mitomycin A is a member of the mitomycin family and its structure has been determined by X-ray analyses using heavy-atom derivatives, *i.e. N*-brosylmitomycin A (Tulinsky & van den Hende, 1967) and *N*-(*p*-bromobenzoyl)mitomycin A (I) (Hirayama & Shirahata, 1987). The aziridine ring is believed to play an important role in the antitumor activities of mitomycins. The aziridine rings in both heavy-atom derivatives are modified by large substituents and it is probable that these influence the inherent structure around the ring. To disclose the intrinsic structure of mitomycin A we have undertaken an X-ray analysis of native mitomycin A (II).

Experimental. Deep-violet crystals from chloroform, dimensions $0{\cdot}30 \times 0{\cdot}20 \times 0{\cdot}20$ mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions from setting angles

* To whom correspondence should be addressed.

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Table 1. *Positional parameters and equivalent isotropic temperature factors of non-*H *atoms with e.s.d.'s in parentheses*

The atoms of molecule *B* are asterisked. O*W* denotes the water molecule.

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}(Å^2)
O(5)	0·6818 (1)	0·3691 (3)	0·9766 (3)	5·01 (6)
O(7)	0·7073 (2)	0·2556 (3)	1·4844 (3)	5·28 (6)
O(8)	0·8398 (2)	0·3792 (3)	1·5065 (3)	4·57 (5)
O(9*a*)	0·9041 (1)	0·6727 (2)	1·0599 (3)	3·88 (5)
O(10)	1·0430 (1)	0·4901 (3)	1·2669 (3)	3·93 (5)
O(10*a*)	1·0481 (2)	0·5885 (4)	1·4921 (3)	6·16 (7)
N(1)	0·9460 (2)	0·3319 (3)	0·9564 (4)	4·06 (6)
N(4)	0·8269 (1)	0·4854 (3)	1·0073 (3)	3·41 (5)
N(10*a*)	1·1358 (2)	0·6246 (3)	1·3311 (4)	4·53 (7)
C(1)	0·9540 (2)	0·4745 (4)	0·9673 (4)	3·48 (6)
C(2)	0·9104 (2)	0·4148 (4)	0·8381 (4)	4·29 (7)
C(3)	0·8278 (2)	0·4318 (4)	0·8553 (4)	4·29 (7)
C(4*a*)	0·7932 (2)	0·4277 (3)	1·1189 (4)	3·11 (6)
C(5)	0·7178 (2)	0·3680 (4)	1·0968 (4)	3·72 (7)
C(6*a*)	0·6143 (2)	0·2405 (5)	1·2056 (6)	5·7 (1)
C(6)	0·6883 (2)	0·308	1·2290 (5)	4·07 (7)
C(7*a*)	0·6698 (3)	0·3331 (7)	1·5829 (5)	6·3 (1)
C(7)	0·7291 (2)	0·3165 (4)	1·3595 (4)	3·84 (7)
C(8)	0·8048 (2)	0·3805 (3)	1·3824 (4)	3·40 (6)
C(8*a*)	0·8330 (2)	0·4357 (3)	1·2531 (4)	3·20 (6)
C(9*b*)	0·8797 (3)	0·7289 (4)	0·9199 (5)	5·14 (9)
C(9*a*)	0·9012 (2)	0·5351 (3)	1·0672 (3)	3·12 (5)
C(9)	0·9073 (2)	0·5027 (3)	1·2371 (3)	3·12 (6)
C(10)	0·9753 (2)	0·4232 (4)	1·2972 (4)	3·95 (7)
C(10*a*)	1·0741 (2)	0·5711 (4)	1·3732 (4)	3·91 (7)
O(5)*	1·4587 (2)	0·0689 (5)	0·5636 (3)	8·62 (9)
O(7)*	1·4935 (2)	0·0434 (4)	1·0771 (3)	6·33 (8)
O(8)*	1·3594 (2)	0·1887 (4)	1·0866 (3)	6·28 (8)
O(9*a*)*	1·1856 (2)	0·2212 (4)	0·5983 (4)	6·77 (7)
O(10)*	1·2077 (2)	0·4732 (3)	0·8328 (4)	6·56 (7)
O(10*a*)*	1·2122 (2)	0·5818 (4)	1·0446 (5)	8·18 (9)
N(1)*	1·3374 (2)	0·4598 (4)	0·5630 (5)	6·44 (9)
N(4)*	1·3185 (2)	0·2039 (3)	0·5772 (3)	4·31 (6)
N(10*a*)*	1·1235 (2)	0·6234 (4)	0·8586 (6)	6·90 (9)
C(1)*	1·2613 (2)	0·4064 (4)	0·5486 (5)	5·36 (9)
C(2)*	1·3104 (3)	0·3885 (6)	0·4286 (5)	6·0 (1)
C(3)*	1·3395 (3)	0·2520 (6)	0·4334 (5)	6·0 (1)
C(4*a*)*	1·3647 (2)	0·1667 (4)	0·6941 (4)	3·73 (6)
C(5)*	1·4362 (2)	0·0972 (5)	0·6832 (4)	4·99 (8)
C(6)*	1·4800 (2)	0·0602 (5)	0·8239 (5)	4·89 (8)
C(6*a*)*	1·5541 (3)	−0·0041 (7)	0·8119 (6)	7·3 (1)
C(7)*	1·4511 (2)	0·0822 (4)	0·9546 (4)	4·23 (7)
C(7*a*)*	1·4658 (3)	0·0032 (7)	1·2082 (5)	7·0 (1)
C(8*a*)*	1·3398 (2)	0·1958 (3)	0·8282 (4)	3·48 (6)
C(8)*	1·3796 (2)	0·1578 (4)	0·9659 (4)	3·86 (7)
C(9*b*)*	1·1542 (4)	0·2138 (7)	0·4595 (6)	8·8 (2)
C(9*a*)*	1·2559 (2)	0·2797 (4)	0·6315 (4)	4·12 (7)
C(9)*	1·2697 (2)	0·2746 (4)	0·8033 (4)	3·87 (7)
C(10)*	1·2760 (2)	0·4046 (4)	0·8815 (5)	5·26 (9)
C(10*a*)*	1·1834 (2)	0·5621 (4)	0·9241 (5)	5·83 (9)
O(*W*)	0·9440 (5)	0·900 (1)	0·286 (1)	7·9 (3)

of 25 independent reflections with $35{\cdot}0 \le \theta \le 48{\cdot}0^\circ$. 3982 reflections surveyed in the range $4 \le 2\theta \le 150^\circ$; $-22 \le h \le 22$, $0 \le k \le 12$, $0 \le l \le 12$; 3840 reflections were unique, 3143 observed with $F > 3\sigma(F)$. Three reference reflections monitored periodically showed no significant variation in intensity. Absorption correction was not applied. Structure solved using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier-map recycling. Refinement using the *SDP* package (Frenz, 1985), full-matrix least-squares refinement on *F*, with non-H atoms having anisotropic temperature factors. Most of the H atoms were located from a difference Fourier synthesis but were not refined. $w = 4F_o^2/[(I_o)^2 + (0{\cdot}04I_o)^2]^{1/2}$/Lp, final $R = 0{\cdot}053$, $wR = 0{\cdot}069$, $S = 2{\cdot}74$, maximum shift/e.s.d. in the final least-squares cycle of 0·01, maximum peak in the final difference map 0·49 (6) e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Final fractional coordinates and equivalent *B* values are listed in Table 1.*

Discussion. The chemical structure and numbering system are shown in Fig. 1. Bond lengths, angles and selected torsion angles are shown in Table 2. The structures of the two crystallographically independent molecules are shown in Fig. 2 (Johnson, 1976). The major differences in these two molecules are seen in the conformations of the methoxy group at C(7) and the carbamoyl group. The conformations of the methoxy groups at C(7) and C(9*a*) and the carbamoyl group are remarkably different from those of (I). The torsion angles around the aziridine rings are essentially in the same region in (I) and (II) but significant differences are observed in the angles C(9)—C(9*a*)—C(1)—N(1), C(9)—C(9*a*)—C(1)—C(2), N(4)—C(3)—C(2)—N(1) and N(4)—C(3)—C(2)—C(1). It is noteworthy that although the value of the torsion angle C(1)—C(9*a*)—O(9*a*)—C(9*b*) is within the *gauche* region in both molecules of (II); it is in the *trans* region in (I), mitomycin C (Arora, 1979), mitomycin C dihydrate (Ogawa, Nomura, Fujiwara & Tomita, 1979) and *N*-brosylmitomycin A (Tulinsky & van den Hende, 1967).

The nonplanarity of the benzoquinone ring which was found in the crystal structure of mitomycin C (Arora, 1979) is also observed in (II). The deviations (Å) of the atoms attached to the benzene ring from the least-squares plane defined by C(4*a*), C(5), C(6), C(7), C(8) and C(8*a*) are as follows: O(5) −0·067 (3) [0·059 (5)], O(8) 0·040 (3) [−0·101 (4)], N(4) 0·004 (3) [0·083 (4)], C(9) 0·031 (3) [−0·105 (4)],

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52011 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

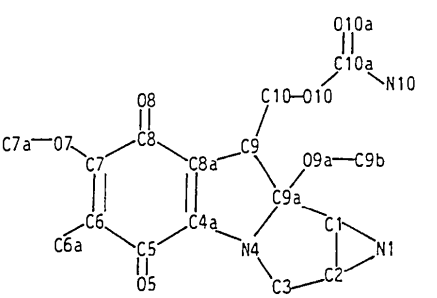

Fig. 1. Atomic notation of mitomycin A.

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°)

	Molecule *A*	Molecule *B*
O(5)—C(5)	1·212 (4)	1·222 (5)
O(7)—C(7)	1·378 (5)	1·345 (5)
O(7)—C(7*a*)	1·412 (6)	1·388 (6)
O(8)—C(8)	1·234 (4)	1·222 (5)
O(9*a*)—C(9*a*)	1·427 (4)	1·405 (5)
O(9*a*)—C(9*b*)	1·424 (5)	1·329 (7)
O(10)—C(10)	1·443 (4)	1·448 (5)
O(10)—C(10*a*)	1·355 (4)	1·335 (6)
O(10*a*)—C(10*a*)	1·221 (5)	1·179 (6)
N(1)—C(1)	1·486 (5)	1·464 (6)
N(1)—C(2)	1·470 (5)	1·465 (6)
N(4)—C(3)	1·482 (5)	1·471 (5)
N(4)—C(4*a*)	1·358 (4)	1·338 (4)
N(4)—C(9*a*)	1·482 (4)	1·486 (5)
N(10)—C(10*a*)	1·322 (5)	1·337 (6)
C(1)—C(2)	1·479 (5)	1·467 (7)
C(1)—C(9*a*)	1·503 (5)	1·518 (6)
C(2)—C(3)	1·510 (5)	1·506 (8)
C(4*a*)—C(5)	1·480 (4)	1·478 (6)
C(4*a*)—C(8*a*)	1·352 (4)	1·362 (5)
C(5)—C(6)	1·484 (5)	1·482 (6)
C(6)—C(6*a*)	1·496 (5)	1·496 (7)
C(6)—C(7)	1·331 (5)	1·350 (6)
C(7)—C(8)	1·506 (5)	1·511 (5)
C(8*a*)—C(8)	1·431 (5)	1·432 (5)
C(8*a*)—C(9)	1·518 (4)	1·496 (5)
C(9*a*)—C(9)	1·565 (4)	1·550 (6)
C(9)—C(10)	1·527 (5)	1·520 (6)
C(7*a*)—O(7)—C(7)	116·1 (4)	124·9 (3)
C(9*a*)—O(9*a*)—C(9*b*)	116·1 (3)	121·3 (4)
C(10*a*)—O(10)—C(10)	117·4 (3)	117·6 (4)
C(1)—N(1)—C(2)	60·0 (2)	60·1 (3)
C(3)—N(4)—C(4*a*)	124·4 (3)	127·2 (3)
C(3)—N(4)—C(9*a*)	112·6 (3)	111·9 (3)
C(4*a*)—N(4)—C(9*a*)	109·0 (2)	108·8 (3)
N(1)—C(1)—C(2)	59·4 (2)	60·0 (3)
N(1)—C(1)—C(9*a*)	113·1 (3)	112·5 (3)
C(2)—C(1)—C(9*a*)	109·4 (3)	108·8 (4)
N(1)—C(2)—C(1)	60·5 (2)	59·9 (3)
N(1)—C(2)—C(3)	111·0 (3)	111·2 (4)
C(1)—C(2)—C(3)	108·9 (3)	109·0 (4)
N(4)—C(3)—C(2)	103·3 (3)	103·2 (4)
N(4)—C(4*a*)—C(5)	122·9 (3)	124·4 (3)
N(4)—C(4*a*)—C(8*a*)	113·9 (3)	114·1 (3)
C(5)—C(4*a*)—C(8*a*)	123·1 (3)	121·5 (3)
O(5)—C(5)—C(4*a*)	121·5 (3)	122·1 (3)
O(5)—C(5)—C(6)	121·6 (3)	120·3 (4)
C(4*a*)—C(5)—C(6)	116·9 (3)	117·6 (3)
C(5)—C(6)—C(6*a*)	117·2 (3)	117·2 (4)
C(5)—C(6)—C(7)	118·6 (3)	119·4 (4)
C(6*a*)—C(6)—C(7)	124·3 (4)	123·4 (4)
O(7)—C(7)—C(6)	121·4 (3)	115·8 (3)
O(7)—C(7)—C(8)	114·0 (3)	121·2 (3)
C(6)—C(7)—C(8)	124·4 (3)	122·7 (3)
C(4*a*)—C(8*a*)—C(8)	120·7 (3)	122·2 (3)
C(4*a*)—C(8*a*)—C(9)	109·7 (3)	109·0 (3)
C(8)—C(8*a*)—C(9)	129·5 (3)	128·8 (3)
O(8)—C(8)—C(7)	119·8 (3)	121·1 (3)
O(8)—C(8)—C(8*a*)	123·9 (3)	122·5 (4)
C(7)—C(8)—C(8*a*)	116·3 (3)	116·3 (3)
O(9*a*)—C(9*a*)—N(4)	111·4 (3)	112·9 (3)
O(9*a*)—C(9*a*)—C(1)	111·2 (3)	111·3 (3)
O(9*a*)—C(9*a*)—C(9)	105·1 (2)	104·9 (3)
N(4)—C(9*a*)—C(1)	103·2 (2)	102·2 (3)
N(4)—C(9*a*)—C(9)	105·3 (2)	104·8 (3)
C(1)—C(9*a*)—C(9)	120·5 (3)	120·7 (3)
C(8*a*)—C(9)—C(9*a*)	102·0 (2)	103·0 (3)
C(8*a*)—C(9)—C(10)	113·4 (3)	112·8 (3)
C(9*a*)—C(9)—C(10)	116·6 (3)	115·7 (3)
O(10)—C(10*a*)—O(10*a*)	123·3 (3)	123·3 (4)
O(10)—C(10*a*)—N(10)	110·9 (3)	110·3 (4)
O(10*a*)—C(10*a*)—N(10)	125·8 (4)	126·4 (5)
O(10)—C(10)—C(9)	109·3 (3)	105·5 (3)
C(8)—C(7)—O(7)—C(7*a*)	87·0 (4)	35·9 (7)
C(8*a*)—C(9)—C(10)—O(10)	175·7 (3)	173·8 (3)
C(9)—C(10)—O(10)—C(10*a*)	91·9 (4)	152·9 (4)
C(9*a*)—C(9)—C(10)—O(10)	57·8 (4)	55·6 (4)
C(9)—C(9*a*)—O(9*a*)—C(9*b*)	−163·8 (3)	177·1 (5)
C(9)—C(9*a*)—C(1)—N(1)	63·8 (4)	65·1 (5)
C(9)—C(9*a*)—C(1)—C(2)	127·9 (3)	129·6 (4)
C(9*a*)—C(1)—C(2)—C(3)	−2·0 (4)	−1·6 (5)
C(9*a*)—C(1)—N(1)—C(2)	99·6 (3)	99·5 (4)

Table 2 (*cont.*)

	Molecule *A*	Molecule *B*
N(4)—C(3)—C(2)—N(1)	56·9 (4)	52·6 (5)
N(4)—C(3)—C(2)—C(1)	−7·9 (4)	−11·6 (5)
C(10)—O(10)—C(10*a*)—O(10*a*)	0·9 (5)	−4·6 (7)
C(10)—O(10)—C(10*a*)—N(10)	179·5 (3)	175·2 (4)
C(8)—C(8*a*)—C(9)—C(10)	52·7 (5)	55·0 (5)

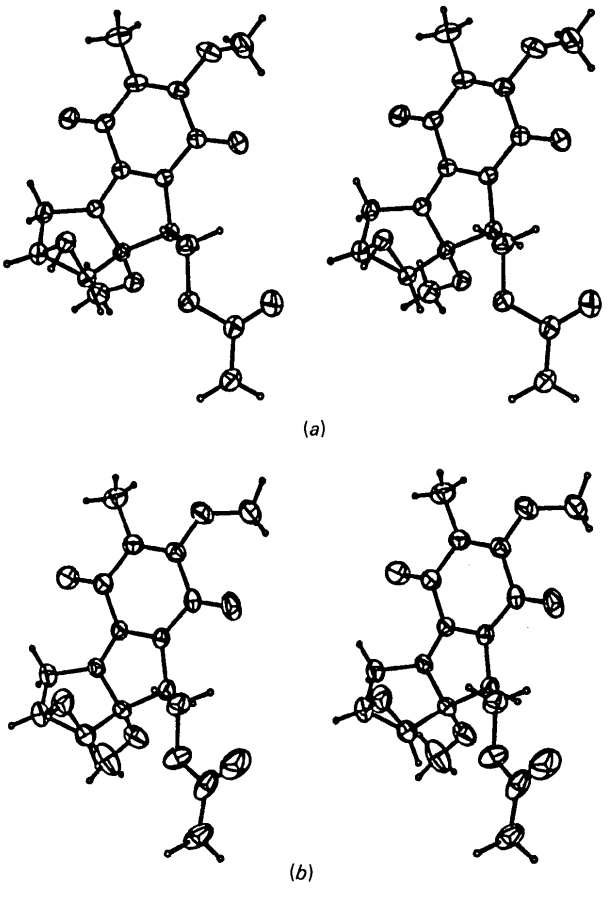

Fig. 2. Stereoview of (*a*) molecule *A* and (*b*) molecule *B* with thermal ellipsoids at 30% probability.

O(7) 0·113 (3) [0·018 (4)], C(6*a*) 0·072 (5) [−0·087 (7)] (the values of molecule *B* are in square brackets). The deviations (Å) of O(5), O(8) and O(7) from the corresponding planes in (I) are 0·106 (4) [−0·095 (4)], 0·075 (4) [−0·056 (5)] and −0·025 (4) [0·006 (5)], respectively. In (II) O(5) and O(8) are located on opposite sides of the least-squares plane, but in (I) and mitomycin C (Arora, 1979) these atoms are on the same side.

Significant differences in the bond lengths and angles around the substituent groups whose conformations are different in the two independent molecules are observed. Although the bond angles around the aziridine ring are almost the same, the bond lengths N(1)—C(1), C(1)—C(2) and

N(1)—C(2) differ significantly. All these bonds in molecule *A* are significantly longer than those in molecule *B*. The average bond lengths in the aziridine ring of (II) are similar to those of (I). In (I) the non-bonded intramolecular N(1)···N(4) distances of the two molecules are significantly different.

The corresponding bond lengths around the benzoquinone ring of crystallographically independent molecules in (II) and mitomycin C are rather variable. This implies that the electronic character around the ring is relatively easily influenced by the environment. However, it is useful to average the corresponding bond lengths of independent molecules in (II) and mitomycin C and compare them in order to understand the effect of substituent groups at the C(7) position [*O*-methyl and amino group in (II) and mitomycin C, respectively] on the electronic character of the benzoquinone ring. The comparison reveals that in (II) bonds C(6)—C(7), O(5)—C(5) and O(8)—C(8) are significantly shorter and C(5)—C(6) and C(8)—C(8*a*) remarkably longer than the corresponding bonds in mitomycin C (Arora, 1979).

Although there are no intramolecular hydrogen bonds, the crystal structure is built up by six intermolecular hydrogen bonds as follows: O(7)···N(1)* $(2-x, -\frac{1}{2}+y, 2-z)$, O(9*a*)···N(1) $(2-x, \frac{1}{2}+y, 2-z)$, N(1)···N(10)* $(2-x, -\frac{1}{2}+y, 2-z)$, N(1)···O(*w*) $(2-x, -\frac{1}{2}+y, 1-z)$, O(10)*···O(*w*) $(2-x, -\frac{1}{2}+y, 1-z)$ and N(10*···O(*w*) $(2-x, -\frac{1}{2}+y, 1-z)$. Thus, *A* and *B* molecules are connected to make a dimer through O(7)···N(1)* and N(1)···N(10)* hydrogen bonds, the latter involving the water molecule. *A* molecules are connected to each other through O(9*a*)···N(1) hydrogen bonds but there is no intermolecular hydrogen bond between *B* molecules.

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Structures of Colchicine Analogues. II. 2′,3′,4′-Trimethoxybiphenyl-3(and -4)-carboxylic Acid Methyl Esters

BY M. F. MACKAY AND L. H. SANDS

Department of Chemistry, La Trobe University, Bundoora, Victoria 3083, *Australia*

E. LACEY

CSIRO, Division of Animal Health, McMaster Laboratory, Private Bag No. 1, *PO Glebe, NSW* 2037, *Australia*

AND P. BURDEN

Pharmacy Department, University of Sydney, Sydney, NSW 2007, *Australia*

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Abstract. $C_{17}H_{18}O_5$, $M_r = 302{\cdot}3$, λ(Cu $K\alpha$) = 1·5418 Å, $T = 289$ (1) K. Methyl 2′,3′,4′-trimethoxybiphenyl-3-carboxylate (IV): monoclinic, $P2_1/c$, $a = 21{\cdot}208$ (2), $b = 5{\cdot}130$ (1), $c = 14{\cdot}172$ (2) Å, $\beta = 92{\cdot}13$ (1)°, $V = 1540{\cdot}8$ (7) Å^3, $Z = 4$, D_m(flotation) = 1·30 (1), $D_x = 1{\cdot}303$ Mg m^{-3}, $\mu = 0{\cdot}71$ mm^{-1}, $F(000) = 640$, final $R = 0{\cdot}046$ for 2282 observed data. Methyl 2′,3′,4′-trimethoxybiphenyl-4-carboxylate (V): orthorhombic, $Pna2_1$, $a = 9{\cdot}499$ (1), $b = 22{\cdot}987$ (1), $c = 14{\cdot}038$ (1) Å, $V = 3065{\cdot}2$ (3) Å^3, $Z = 8$, D_m(flotation) = 1·30 (1), $D_x = 1{\cdot}309$ Mg m^{-3}, $\mu = 0{\cdot}71$ mm^{-1}, $F(000) = 1280$, final $R = 0{\cdot}045$ for 2630 observed data. The two independent molecules of (V) adopt similar conformations with the dihedral angle

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