$C_{11}H_{21}O_4P.2H_2O$

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

Molecule 1		Molecule 2	
P(1)—O(1)	1·485 (3)	P(2)—O(21)	1·485 (3)
P(1)—O(2)	1·569 (3)	P(2)—O(22)	1·564 (3)
P(1)—C(1)	1·842 (4)	P(2)—C(21)	1·842 (4)
P(1)—C(8)	1·810 (4)	P(2)—C(28)	1·813 (4)
O(3)—C(1)	1·444 (4)	O(23)—C(21)	1·449 (4)
O(4)—C(8)	1·434 (4)	O(24)—C(28)	1·441 (4)
C(1)—C(2)	1·515 (5)	C(21)—C(22)	1·520 (5)
C(1)—C(9)	1·526 (5)	C(21)—C(29)	1·521 (5)
C(2)—C(3)	1·550 (5)	C(22)—C(23)	1·548 (5)
C(3)—C(4)	1·547 (6)	C(23)—C(24)	1·538 (6)
C(3)—C(10)	1·535 (6)	C(23)—C(30)	1·533 (6)
C(3)—C(11)	1·536 (6)	C(23)—C(31)	1·538 (6)
C(4)—C(5)	1·540 (5)	C(24)—C(25)	1·540 (5)
C(4)—C(8)	1·526 (5)	C(24)—C(28)	1·531 (5)
C(5)—C(6)	1·523 (7)	C(25)—C(26)	1·523 (7)
C(6)—C(7)	1·534 (6)	C(26)—C(27)	1·539 (6)
C(7)—C(8)	1·524 (5)	C(27)—C(28)	1·540 (5)
O(2)—P(1)—O(1)	113·7 (2)	O(22)—P(2)—O(21)	113·5 (2)
C(1)—P(1)—O(1)	113·2 (2)	C(21)—P(2)—O(21)	113·5 (2)
C(1)—P(1)—O(2)	106·2 (2)	C(21)—P(2)—O(22)	106·1 (2)
C(8)—P(1)—O(1)	113·7 (2)	C(28)—P(2)—O(21)	113·7 (2)
C(8)—P(1)—O(2)	104·1 (2)	C(28)—P(2)—O(22)	104·5 (2)
C(8)—P(1)—C(1)	105·0 (2)	C(28)—P(2)—C(21)	104·7 (2)
O(3)—C(1)—P(1)	106·7 (2)	O(23)—C(21)—P(2)	106·4 (2)
C(2)—C(1)—P(1)	110·4 (2)	C(22)—C(21)—P(2)	110·4 (3)
C(2)—C(1)—O(3)	104·5 (3)	C(22)—C(21)—O(23)	104·6 (3)
C(9)—C(1)—P(1)	109·8 (2)	C(29)—C(21)—P(2)	110·3 (3)
C(9)—C(1)—O(3)	110·1 (3)	C(29)—C(21)—O(23)	109·4 (3)
C(9)—C(1)—C(2)	115·0 (3)	C(29)—C(21)—C(22)	115·2 (3)
C(3)—C(2)—C(1)	121·7 (3)	C(23)—C(22)—C(21)	121·4 (3)
C(4)—C(3)—C(2)	108·7 (3)	C(24)—C(23)—C(22)	108·6 (3)
C(10)—C(3)—C(2)	112·6 (4)	C(30)—C(23)—C(22)	112·7 (4)
C(10)—C(3)—C(4)	114·2 (3)	C(30)—C(23)—C(24)	114·4 (3)
C(11)—C(3)—C(2)	106·4 (4)	C(31)—C(23)—C(22)	105·7 (3)
C(11)—C(3)—C(4)	107·5 (4)	C(31)—C(23)—C(24)	107·7 (4)
C(11)—C(3)—C(10)	107·1 (4)	C(31)—C(23)—C(30)	107·2 (4)
C(5)—C(4)—C(3)	119·3 (3)	C(25)—C(24)—C(23)	119·3 (4)
C(8)—C(4)—C(3)	117·9 (3)	C(28)—C(24)—C(23)	118·3 (3)
C(8)—C(4)—C(5)	101·9 (3)	C(28)—C(24)—C(25)	101·4 (3)
C(6)—C(5)—C(4)	104·1 (4)	C(26)—C(25)—C(24)	103·5 (4)
C(7)—C(6)—C(5)	106·8 (4)	C(27)—C(26)—C(25)	107·3 (4)
C(8)—C(7)—C(6)	105·1 (4)	C(28)—C(27)—C(26)	104·3 (4)
O(4)—C(8)—P(1)	106·9 (2)	O(24)—C(28)—P(2)	106·0 (2)
C(4)—C(8)—P(1)	110·5 (3)	C(24)—C(28)—P(2)	111·5 (2)
C(4)—C(8)—O(4)	108·8 (3)	C(24)—C(28)—O(24)	108·5 (3)
C(7)—C(8)—P(1)	116·1 (3)	C(27)—C(28)—P(2)	116·5 (3)
C(7)—C(8)—O(4)	111·2 (3)	C(27)—C(28)—O(24)	111·1 (3)
C(7)—C(8)—C(4)	103·1 (3)	C(27)—C(28)—C(24)	103·2 (3)

Fig. 1. CHEMGRAF (Davies, 1981) drawing of the asymmetric unit content showing the numbering scheme.

References

BARTCZAK, T. & YAGBASAN, R. (1991). Acta Cryst. C47, 1750–1752

BODALSKI, R., KRAWCZYK, H. & MAJEWSKI, P. (1987a). Unpublished results.

BODALSKI, R., KRAWCZYK, H. & MAJEWSKI, P. (1987b). Private communications.

CARRUTHERS, J. R. & WATKIN, D. J. (1979). Acta Cryst. A35, 695–699.

CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.

DAVIES, E. K. (1981). CHEMGRAF. User Guide. Chemical Crystallography Laboratory, Univ. of Oxford, England.

SHELDRICK, G. M. (1985). SHELXS86. Crystallographic Computing 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

VIĆKÓVIĆ, I. (1988). CSU. Crystal Structure Utility. Univ. of Zagreb, Yugoslavia.

WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, P. W. (1985). CRYSTALS. User Guide. Chemical Crystallography Laboratory, Univ. of Oxford, England.

ture of 1,2,7a-trihydroxy-2-methylperhydro-1-phosphaindene 1-oxide has been reported proviously (Bartczak & Yagbasan, 1991).

Structure of the Triclinic Modification of 17β-Hydroxy-19-nor-7α-methyl-17α-pregn-5(10)-en-20-yn-3-one (ORG OD14)

By A. SCHOUTEN AND J. A. KANTERS

Department of Crystal and Structural Chemistry, Bijvoet Center for Biomolecular Research, Rijksuniversiteit Utrecht, Transitorium 3, Padualaan 8, 3584 CH Utrecht, The Netherlands

Abstract. (I) $C_{21}H_{28}O_2$, $M_r = 312·45$, triclinic, $P1$, $a = 6·542$ (2), $b = 6·6773$ (8), $c = 10·287$ (2) Å, $\alpha = 87·05$ (1), $\beta = 80·09$ (2), $\gamma = 79·17$ (2)°, $V = 434·7$ (2) Å^3, $Z = 1$, $D_x = 1·194$ g cm^{-3}, λ(Cu Kα) = 1·54184 Å, $\mu = 5·5$ cm^{-1}, $F(000) = 170$, room temperature, $R = 0·066$ for 1495 reflections with $I \geq 2·5\sigma(I)$. The A ring has a 2α,3β half-chair conformation, the B ring a 7α,8β half-chair conformation.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters and their e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} (Å^2)
O(3)	0·4433 (5)	1·3391 (5)	0·8133 (3)	0·069 (1)
O(17)	1·1855 (4)	0·7298 (4)	−0·0775 (2)	0·0552 (8)
C(1)	0·3937 (5)	1·0935 (6)	0·5428 (3)	0·050 (1)
C(2)	0·2815 (6)	1·1475 (6)	0·6840 (4)	0·056 (1)
C(3)	0·4426 (6)	1·1761 (6)	0·7642 (3)	0·052 (1)
C(4)	0·6092 (7)	0·9953 (6)	0·7739 (3)	0·055 (1)
C(5)	0·6884 (5)	0·8897 (5)	0·6433 (3)	0·0436 (9)
C(6)	0·8827 (6)	0·7287 (5)	0·6453 (3)	0·051 (1)
C(7)	0·9406 (5)	0·5948 (5)	0·5243 (3)	0·0430 (8)
C(8)	0·9193 (5)	0·7351 (5)	0·4030 (3)	0·0381 (8)
C(9)	0·6904 (5)	0·8447 (5)	0·4048 (3)	0·0387 (8)
C(10)	0·5968 (5)	0·9410 (5)	0·5372 (3)	0·0411 (8)
C(11)	0·6781 (6)	0·9932 (5)	0·2850 (3)	0·0467 (9)
C(12)	0·7759 (5)	0·8883 (5)	0·1530 (3)	0·0446 (9)
C(13)	1·0028 (5)	0·7790 (4)	0·1529 (3)	0·0406 (9)
C(14)	1·0028 (5)	0·6285 (4)	0·2728 (3)	0·0387 (8)
C(15)	1·2233 (5)	0·4992 (6)	0·2517 (3)	0·052 (1)
C(16)	1·2851 (5)	0·4847 (6)	0·1015 (3)	0·050 (1)
C(17)	1·1051 (5)	0·6256 (5)	0·0406 (3)	0·0432 (9)
C(18)	1·1485 (7)	0·9316 (6)	0·1544 (4)	0·054 (1)
C(19)	0·8088 (8)	0·4265 (6)	0·5357 (4)	0·062 (1)
C(20)	0·9545 (5)	0·5057 (5)	0·0064 (3)	0·0437 (8)
C(21)	0·8381 (6)	0·4150 (6)	−0·0299 (4)	0·054 (1)

The *C* ring adopts a slightly distorted chair conformation and the *D* ring is a distorted 13β envelope.

Experimental. The title compound (I) was supplied by Organon International, Oss, The Netherlands. Crystallization by dissolving in acetone and pouring into water yielded crystals with monoclinic space group *P*2₁, the cell dimensions of which correspond to those reported earlier by Declercq, van Meerssche & Zeelen (1984). However, cooling a solution in hot toluene afforded crystals of the triclinic modification. A single crystal with dimensions 0·75 × 0·15 × 0·25 mm was selected. Data were collected on an Enraf–Nonius CAD-4 diffractometer using Cu *Kα* radiation. Cell dimensions were determined by least-squares fitting of the angular settings of 23 reflections in the θ range 8–24°. ω–2θ scan mode, ω = (1·40 + 0·15tanθ)°, 3484 reflections measured, 2θ$_{max}$ = 140°, with −7 ≤ *h* ≤ 7, −8 ≤ *k* ≤ 8, −12 ≤ *l* ≤ 12. Equivalent reflections were merged (R_{int} = 0·051) which resulted in 1495 unique reflections having *I* ≥ 2·5σ(*I*). Two periodically measured standard reflections (112 and 120) showed intensity variations of less than 1·5%. Lp corrections, no correction for absorption. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986). H atoms bonded to C(sp^3) atoms were placed at calculated positions and refined riding on their carrier atoms with an overall isotropic temperature factor. The H atom of O(17)—H could not be located, the same holds for the H atom bonded to the C atom of the ethynyl group. This last atom was placed at a calculated position assuming linearity of the C≡C—H frag-

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

O(3)—C(3)	1·224 (5)	C(9)—C(10)	1·516 (4)
O(17)—C(17)	1·438 (4)	C(9)—C(11)	1·545 (4)
C(1)—C(2)	1·538 (5)	C(11)—C(12)	1·541 (4)
C(1)—C(10)	1·509 (5)	C(12)—C(13)	1·526 (5)
C(2)—C(3)	1·490 (6)	C(13)—C(14)	1·550 (4)
C(3)—C(4)	1·478 (6)	C(13)—C(17)	1·559 (4)
C(4)—C(5)	1·514 (5)	C(13)—C(18)	1·523 (5)
C(5)—C(6)	1·505 (5)	C(14)—C(15)	1·522 (5)
C(5)—C(10)	1·333 (4)	C(15)—C(16)	1·532 (4)
C(6)—C(7)	1·522 (4)	C(16)—C(17)	1·562 (5)
C(7)—C(8)	1·531 (4)	C(17)—C(20)	1·477 (5)
C(7)—C(19)	1·528 (6)	C(20)—C(21)	1·175 (5)
C(8)—C(9)	1·536 (5)		
C(8)—C(14)	1·516 (4)		

C(2)—C(1)—C(10)	113·7 (3)	C(5)—C(10)—C(9)	121·3 (3)
C(1)—C(2)—C(3)	108·5 (3)	C(9)—C(11)—C(12)	112·6 (3)
O(3)—C(3)—C(2)	122·8 (4)	C(11)—C(12)—C(13)	112·1 (3)
O(3)—C(3)—C(4)	122·3 (4)	C(12)—C(13)—C(14)	107·7 (3)
C(2)—C(3)—C(4)	114·8 (3)	C(12)—C(13)—C(17)	117·9 (3)
C(3)—C(4)—C(5)	112·5 (3)	C(12)—C(13)—C(18)	110·9 (3)
C(4)—C(5)—C(6)	113·2 (3)	C(14)—C(13)—C(17)	99·2 (2)
C(4)—C(5)—C(10)	122·5 (3)	C(14)—C(13)—C(18)	112·9 (3)
C(6)—C(5)—C(10)	124·3 (3)	C(17)—C(13)—C(18)	107·9 (3)
C(5)—C(6)—C(7)	113·8 (3)	C(8)—C(14)—C(13)	112·5 (2)
C(6)—C(7)—C(8)	107·4 (3)	C(8)—C(14)—C(15)	119·2 (3)
C(6)—C(7)—C(19)	111·6 (3)	C(13)—C(14)—C(15)	104·5 (3)
C(8)—C(7)—C(19)	113·7 (3)	C(14)—C(15)—C(16)	104·4 (3)
C(7)—C(8)—C(9)	111·5 (3)	C(15)—C(16)—C(17)	106·8 (3)
C(7)—C(8)—C(14)	113·9 (3)	O(17)—C(17)—C(13)	111·1 (3)
C(9)—C(8)—C(14)	109·9 (3)	O(17)—C(17)—C(16)	112·1 (3)
C(8)—C(9)—C(10)	111·3 (3)	O(17)—C(17)—C(20)	107·7 (3)
C(8)—C(9)—C(11)	109·8 (3)	C(13)—C(17)—C(16)	102·9 (2)
C(10)—C(9)—C(11)	114·6 (3)	C(13)—C(17)—C(20)	112·4 (3)
C(1)—C(10)—C(5)	122·1 (3)	C(16)—C(17)—C(20)	110·7 (3)
C(1)—C(10)—C(9)	116·6 (3)	C(17)—C(20)—C(21)	175·3 (4)

ment, but not refined. Blocked full-matrix refinement with *SHELX*76 (Sheldrick, 1976), 209 parameters varied, including scale factor, positional and individual anisotropic thermal parameters for the non-H atoms. Refinement on *F* resulted in a final *R* = 0·066 and *wR* = 0·067 with $w = [\sigma^2(F_o) + 0·0134F_o^2]^{-1}$ and *S* = 0·71. The overall *U* value for the H atoms refined to 0·089 Å^2. The average shift to e.s.d. ratio is 0·02, the maximum ratio is 0·08. The final residual density is within ±0·40 e Å^{-3}. Scattering factors were taken from *SHELX*76.

Table 1* gives the atomic coordinates and equivalent isotropic thermal parameters and Table 2 lists bond distances and angles. Fig. 1 shows a perspective view of the molecule with the adopted numbering scheme and Fig. 2 a view of the unit cell along the *b* axis with the O(17)···O(3′)(1 + *x*, −1 + *y*, −1 + *z*) head-to-tail hydrogen bond [distance 2·985 (4) Å].

Related literature. The reported structures of four 3-oxo-5(10)-ene steroids show a variety of *A* ring

Fig. 1. Perspective view of (I) with atom-numbering scheme.

Fig. 2. View of the unit cell down the *b* axis showing the head-to-tail hydrogen bond between O(17) and O(3′).

conformations. In the triclinic modification of (I) the *A* ring is 2α,3β half-chair, whereas in the monoclinic form the *A* rings of the two independent molecules adopt 2α,3β and 2β,3α half-chair conformations respectively (Schouten & Kanters, 1990; Declercq, van Meerssche & Zeelen, 1984).

In norethynodrel, 17β-hydroxy-19-nor-17α-pregn-5(10)-en-20-yn-3-one (McPhail, Luhan, Wong Tschang & Onan, 1977), the *A* rings of the two independent molecules have C(2)-α and C(2)-β envelope conformations, respectively, and in 17β-(iodoacetoxy)estr-5(10)-en-3-one (Sobti, Bordner & Levine, 1971) and 17β-hydroxyestr-5(10)-en-3-one (Sobti, Levine & Bordner, 1972) the *A* rings are also C(2)-α envelopes.

References

DECLERCQ, J.-P., VAN MEERSSCHE, M. & ZEELEN, F. J. (1984). *Recl Trav. Chim. Pays-Bas*, **103**, 145–147.
McPHAIL, A. T., LUHAN, P. A., WONG TSCHANG, P. & ONAN, K. D. (1977). *J. Chem. Soc. Perkin Trans.* 2, pp. 379–383.
SCHOUTEN, A. & KANTERS, J. A. (1990). Unpublished work.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination, Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
SOBTI, R. R., BORDNER, J. & LEVINE, S. G. (1971). *J. Am. Chem. Soc.* **93**, 5588–5590.
SOBTI, R. R., LEVINE, S. G. & BORDNER, J. (1972). *Acta Cryst.* **B28**, 2292–2297.

Structure de la Bétaïne du Carboxyméthyl-1 Méthylamino-4 Triazolium-1,2,4

PAR L. DUPONT, S. ENGLEBERT ET O. DIDEBERG

Laboratoire de Cristallographie, Institut de Physique B5, Université de Liège au Sart Tilman, B-4000 Liège, Belgique

ET B. PIROTTE ET J. DELARGE

Laboratoire de Chimie Pharmaceutique, Institut de Pharmacie F1, Université de Liège, rue Fusch, 3–5, B-4000 Liège, Belgium

Abstract. 1-Carboxymethyl-4-methylamino-1,2,4-triazolium betaine, $C_5H_8N_4O_2$, $M_r = 156.14$, monoclinic, $P2_1/a$, $a = 7.496\,(1)$, $b = 12.898\,(1)$, $c = 7.533\,(1)$ Å, $\beta = 101.95\,(1)°$, $V = 712.5\,(1)$ Å^3, $Z = 4$, $D_x = 1.455$ Mg m^{-3}, $\lambda(\text{Cu } K\bar{\alpha}) = 1.5418$ Å, $\mu = 1.0$ mm^{-1}, $F(000) = 328$, $T = 294\,(1)$ K, $R = 0.078$ for 777 observed reflections. The triazolium ring is planar [maximum deviation 0.006 (4) Å] and C(2) and N(4) are significantly out of this plane [-0.081 (4) and 0.101 (4) Å respectively]. There is one N—H···O hydrogen bond 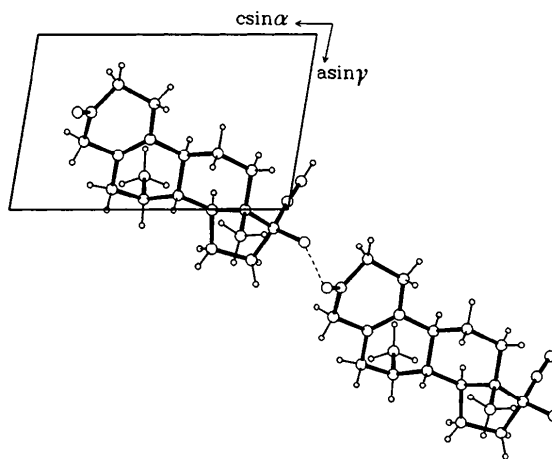with N···O 2.892 (4), N—H 1.14 (4), H···O 1.76 (4) Å and N—H—O angle