Acta Cryst. (1994). C50, 964–965

3-Methoxy-16,16-dimethyl-5α-androstan-17-one

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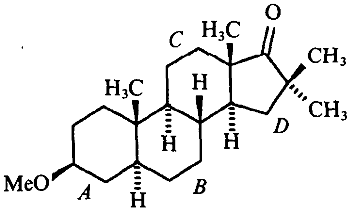
(*Received* 19 *July* 1993; *accepted* 9 *November* 1993)

Abstract

The structure of the title compound, $C_{22}H_{36}O_2$, is reported.

Comment

The synthesis of the title compound, (I), has been reported in an earlier paper (Wölfling, Schneider & Dombi, 1988).



(I)

The *A* and *B*, *B* and *C*, and *C* and *D* rings are *trans* fused. Rings *A*, *B* and *C* adopt chair conformations, whereas ring *D* shows a conformation midway between a 14α-envelope and a 13β,14α-half chair. For structures of other 5α-androstane derivatives see Duax & Norton (1975) and Griffin, Duax & Weeks (1984).

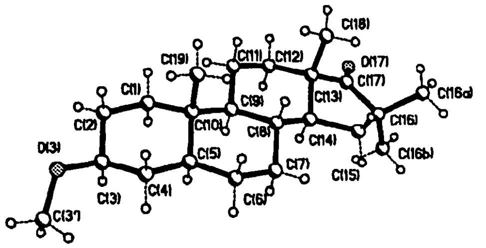


Fig. 1. The atomic numbering scheme for the title compound.

Experimental

Crystal data

$C_{22}H_{36}O_2$
$M_r = 332.51$
Orthorhombic
$P2_12_12_1$
$a = 6.5620\ (10)$ Å
$b = 13.583\ (3)$ Å
$c = 22.488\ (4)$ Å
$V = 2004.4\ (6)$ Å^3
$Z = 4$
$D_x = 1.102$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71072$ Å
Cell parameters from 48 reflections
$\theta = 10$–$12.5°$
$\mu = 0.068$ mm^{-1}
$T = 293\ (2)$ K
Needles
$0.50 \times 0.40 \times 0.40$ mm
Colorless

Data collection

Stoe AED-2 four-circle diffractometer
Profile fitting collection method
Absorption correction: none
2055 measured reflections
1769 independent reflections
1574 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0108$
$\theta_{\rm max} = 22.50°$
$h = -1 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -24 \rightarrow 24$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0363$
$wR(F^2) = 0.0960$
$S = 0.968$
1768 reflections
218 parameters
H-atom parameters not refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0610P)^2 + 0.5000P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.009$
$\Delta\rho_{\rm max} = 0.119$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.116$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001\chi \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.012\ (2)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	$U_{\rm eq}$
C1	−0.1710 (6)	−0.5081 (2)	−0.17046 (13)	0.0542 (8)
C2	−0.1458 (6)	−0.6103 (2)	−0.14299 (14)	0.0601 (9)
C3	0.0256 (6)	−0.6124 (2)	−0.09872 (14)	0.0588 (9)
O3	0.0338 (5)	−0.7096 (2)	−0.07388 (11)	0.0802 (8)
C3′	0.2192 (9)	−0.7317 (3)	−0.0455 (2)	0.111 (2)
C4	−0.0051 (7)	−0.5341 (2)	−0.05120 (13)	0.0596 (9)
C5	−0.0294 (5)	−0.4321 (2)	−0.07904 (12)	0.0485 (8)
C6	−0.0393 (7)	−0.3510 (2)	−0.03240 (13)	0.0610 (10)
C7	−0.0396 (6)	−0.2493 (2)	−0.06111 (12)	0.0551 (9)
C8	−0.2041 (5)	−0.2385 (2)	−0.10876 (12)	0.0454 (8)
C9	−0.1922 (5)	−0.3235 (2)	−0.15461 (12)	0.0433 (7)
C10	−0.2058 (5)	−0.4262 (2)	−0.12412 (12)	0.0434 (7)
C11	−0.3390 (6)	−0.3094 (2)	−0.20691 (12)	0.0546 (9)
C12	−0.3254 (6)	−0.2081 (2)	−0.23612 (13)	0.0566 (9)
C13	−0.3512 (5)	−0.1277 (2)	−0.18950 (13)	0.0480 (8)
C14	−0.1893 (5)	−0.1407 (2)	−0.14117 (11)	0.0445 (7)
C15	−0.1946 (6)	−0.0435 (2)	−0.10730 (13)	0.0556 (9)
C16	−0.2234 (5)	0.0359 (2)	−0.15601 (13)	0.0525 (8)
C16*A*	−0.3700 (7)	0.1181 (2)	−0.1383 (2)	0.0787 (11)
C16*B*	−0.0203 (6)	0.0820 (2)	−0.1742 (2)	0.0668 (10)

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

C17	−0.3084 (5)	−0.0230 (2)	−0.20887 (14)	0.0524 (8)
O17	−0.3305 (4)	0.0101 (2)	−0.25857 (10)	0.0719 (7)
C18	−0.5715 (5)	−0.1271 (3)	−0.1660 (2)	0.0679 (10)
C19	−0.4161 (5)	−0.4413 (2)	−0.09522 (15)	0.0605 (9)

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.528 (4)	C9—C10	1.557 (4)
C1—C10	1.542 (4)	C10—C19	1.539 (5)
C2—C3	1.502 (5)	C11—C12	1.527 (4)
C3—O3	1.435 (4)	C12—C13	1.524 (4)
C3—C4	1.521 (4)	C13—C17	1.513 (4)
O3—C3′	1.406 (5)	C13—C14	1.530 (4)
C4—C5	1.529 (4)	C13—C18	1.539 (5)
C5—C6	1.522 (4)	C14—C15	1.524 (4)
C5—C10	1.541 (4)	C15—C16	1.549 (4)
C6—C7	1.526 (4)	C16—C16*A*	1.527 (5)
C7—C8	1.528 (4)	C16—C16*B*	1.529 (5)
C8—C14	1.518 (4)	C16—C17	1.538 (4)
C8—C9	1.550 (3)	C17—O17	1.213 (3)
C9—C11	1.532 (4)		
C2—C1—C10	113.5 (2)	C1—C10—C9	109.9 (2)
C3—C2—C1	111.5 (3)	C12—C11—C9	113.9 (2)
O3—C3—C2	107.7 (3)	C13—C12—C11	110.1 (2)
O3—C3—C4	112.0 (2)	C17—C13—C12	117.1 (2)
C2—C3—C4	110.7 (3)	C17—C13—C14	100.6 (2)
C3′—O3—C3	114.0 (3)	C12—C13—C14	109.2 (2)
C3—C4—C5	111.1 (2)	C17—C13—C18	105.6 (3)
C6—C5—C4	112.2 (2)	C12—C13—C18	110.1 (3)
C6—C5—C10	112.6 (3)	C14—C13—C18	114.1 (2)
C4—C5—C10	113.3 (3)	C8—C14—C15	121.1 (2)
C5—C6—C7	111.3 (2)	C8—C14—C13	113.4 (2)
C6—C7—C8	112.6 (3)	C15—C14—C13	103.8 (2)
C14—C8—C7	112.1 (2)	C14—C15—C16	104.6 (2)
C14—C8—C9	109.2 (2)	C16*A*—C16—C16*B*	108.6 (3)
C7—C8—C9	111.1 (2)	C16*A*—C16—C17	110.7 (3)
C11—C9—C8	112.7 (2)	C16*B*—C16—C17	108.8 (3)
C11—C9—C10	114.5 (2)	C16*A*—C16—C15	113.7 (3)
C8—C9—C10	111.8 (2)	C16*B*—C16—C15	111.6 (3)
C19—C10—C5	112.9 (2)	C17—C16—C15	103.2 (2)
C19—C10—C1	108.7 (3)	O17—C17—C13	126.2 (3)
C5—C10—C1	107.2 (2)	O17—C17—C16	124.2 (3)
C19—C10—C9	110.9 (3)	C13—C17—C16	109.5 (2)
C5—C10—C9	107.1 (2)		

Data collection: *DIF*4 (Stoe & Cie, 1989*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1989*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992).

This work was supported by the Fonds der Chemischen Industrie and by the Hungarian Research Fund (grant No. OTKA F4308). A research fellowship to JW from the Alexander von Humboldt Foundation is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1056). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 965–967

An Enantiomerically Pure 4-Oxo-α-amino Acid Derivative

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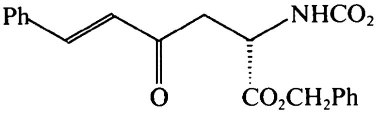
(*Received* 12 *August* 1993; *accepted* 4 *January* 1994)

Abstract

In the molecule of benzyl 2(*S*)-[(*tert*-butoxycarbonyl)amino]-4-oxo-6-phenyl-5(*E*)-hexenoate, $C_{24}H_{27}NO_5$, the amine NH group does not form intramolecular hydrogen bonds with any of the three carbonyl groups present, even the most favourably positioned one, which would give a six-membered ring. Instead, weak intermolecular hydrogen-bonding interactions occur between the NH and CO groups of the amide units in adjacent molecules, linking them into a chain.

Comment

Enantiomerically pure α-amino acids can be prepared in various ways (Williams, 1989). Recent research into methods which do not involve the formation of one of the four bonds to the α centre and which instead retain the chirality already established at this centre, has been successful in producing a wide range of protected 4-oxo-α-amino acids and protected phenylalanine analogues from an organozinc intermediate (Jackson, Wishart, Wood, James & Wythes, 1992). The title compound, (I), is one of these 4-oxo-α-amino acid derivatives. Its structure has been determined in order to confirm the enantiomeric purity and to investigate the hydrogen bonding which may be expected to occur between the amino group and one of the three carbonyl groups within the molecule.



(I)