Table 3. *Selected distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Molecule *A*		Molecule *B*	
Pd1—Cl1	2·380 (2)	Pd2—Cl2	2·389 (2)
Pd1—N1	2·028 (6)	Pd2—N21	2·009 (7)
Pd1—C4	1·937 (9)	Pd2—C24	1·924 (8)
Pd1—C11	2·002 (6)	Pd2—C211	2·036 (6)
N1—C1	1·378 (9)	N21—C21	1·370 (8)
N1—C3	1·311 (8)	N21—C23	1·339 (9)
N2—C2	1·384 (8)	N22—C22	1·373 (8)
N2—C3	1·34 (2)	N22—C23	1·33 (1)
N2—C9	1·461 (9)	N22—C29	1·48 (2)
N3—C4	1·14 (2)	N23—C24	1·16 (1)
N3—C5	1·47 (2)	N23—C25	1·45 (1)
C1—C2	1·35 (1)	C21—C22	1·36 (1)
C3—C12	1·465 (9)	C23—C212	1·458 (8)
Cl1—Pd1—N1	93·0 (1)	Cl2—Pd2—N21	93·0 (2)
Cl1—Pd1—C4	92·1 (2)	Cl2—Pd2—C24	93·2 (3)
Cl1—Pd1—C11	173·7 (2)	Cl2—Pd2—C211	174·0 (2)
N1—Pd1—C4	173·8 (2)	N21—Pd2—C24	173·4 (2)
N1—Pd1—C11	80·8 (3)	N21—Pd2—C211	81·4 (2)
C4—Pd1—C11	94·1 (3)	C24—Pd2—C211	92·3 (3)
Pd1—N1—C1	136·1 (4)	Pd2—N21—C21	138·3 (5)
Pd1—N1—C3	115·4 (5)	Pd2—N21—C23	114·8 (4)
C1—N1—C3	107·9 (6)	C21—N21—C23	106·6 (7)
C2—N2—C3	106·6 (6)	C22—N22—C23	108·7 (7)
C2—N2—C9	124·8 (6)	C22—N22—C29	123·7 (6)
C3—N2—C9	128·5 (5)	C23—N22—C29	127·5 (5)
C4—N3—C5	179·2 (6)	C24—N23—C25	178·9 (7)
N1—C1—C2	107·6 (6)	N21—C21—C22	109·2 (6)
N2—C2—C1	107·4 (7)	N22—C22—C21	105·8 (6)
N1—C3—N2	110·6 (6)	N21—C23—N22	109·7 (5)
N1—C3—C12	116·7 (7)	N21—C23—C212	116·0 (6)
N2—C3—C12	132·7 (6)	N22—C23—C212	134·2 (7)
Pd1—C4—N3	178·8 (6)	Pd2—C24—N23	178·7 (7)
Solvent			
Cl31—C30	1·75 (1)	Cl41—C40	1·73 (1)
Cl32—C30	1·75 (1)	Cl42—C40	1·71 (1)

Experimental. A colorless single crystal of approximate dimensions 0·10 × 0·15 × 0·15 mm was sealed in a capillary. Because the crystals decompose at room temperature all X-ray investigations were performed at 213 K. The systematic absences pointed to space group $P2_1/c$, and this space group was used for all further calculations. In the final full-matrix least-squares refinement all non-H atoms were assigned anisotropic thermal parameters. More details of the intensity data collection, structure solution and refinement are listed in Table 1. Final atomic coordinates are given in Table 2, selected distances and angles in Table 3.* The two independent molecules *A* and *B* in the unit cell have the same structural features. A *SCHAKAL* (Keller, 1988) plot of molecule *A* is shown in Fig. 1, a stereoview of the unit cell in Fig. 2.

Related literature. The structure determination is part of our studies of cyclometallated complexes derived from Schiff-base ligands (Gayoso, Alonso, Vila, Rivero, Hiller & Strähle, 1988; Hiller, Castiñeiras, Vila, Suarez, Pereira & Gayoso, 1986; Pereira, Vila, Gayoso, Gayoso, Hiller & Strähle, 1988; Suarez, Vila, Gayoso, Gayoso, Hiller, Castiñeiras & Strähle, 1986).

We would like to acknowledge a research grant from DAAD (AC) and thank Professor J. Strähle for providing us with facilities.

---

* Lists of structure factors, H-atom positions, bond distances and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51883 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Frenz, B.A. (1978). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography,* edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.

Gayoso, M., Alonso, M., Vila, J. M., Rivero, E. B., Hiller, W. & Strähle, J. (1988). *Acta Cryst.* C**44**, 929–931.

Hiller, W., Castiñeiras, A., Vila, J. M., Suarez, A., Pereira, M. T. & Gayoso, M. (1986). *Acta Cryst.* C**42**, 1136–1138.

International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Keller, E. (1988). *SCHAKAL*. Univ. of Freiburg.

Pereira, M. T., Vila, J. M., Gayoso, E., Gayoso, M., Hiller, W. & Strähle, J.(1988). *J. Coord. Chem.* **18**, 245–252.

Suarez, A., Vila, J. M., Gayoso, E., Gayoso, M., Hiller, W., Castiñeiras, A. & Strähle, J. (1986). *Z. Anorg. Allg. Chem.* **535**, 213–218.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1989). C**45**, 1433–1435

Structure of 3β-Hydroxy-16α-methyl-5-pregnen-20-one

By W. L. Duax, J. F. Griffin, P. D. Strong and Y. Guan

Medical Foundation of Buffalo, Inc., 73 *High Street, Buffalo, NY* 14203, *USA*

(*Received* 13 *February* 1989; *accepted* 4 *April* 1989)

Abstract. $C_{22}H_{34}O_2$, $M_r = 330·52$, triclinic, $P1$, $a = 6·3650$ (5), $b = 12·900$ (1), $c = 6·3365$ (5) Å, $\alpha = 96·488$ (9), $\beta = 107·361$ (9), $\gamma = 98·843$ (8)°, $V = 483·66$ Å^3, $Z = 1$, $D_x = 1·135$ g cm^3, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·655$ cm^{-1}, $F(000) = 182$, $T = 298$ K, $R = 0·043$ for 2587 unique observed reflec-

0108-2701/89/091433-03$03.00

Table 1. *Fractional positional parameters* ($\times 10^4$) *and equivalent isotropic atomic displacement parameters* ($B_{eq} \times 10$ Å^2) *for non*-H *atoms with e.s.d.'s in parentheses*

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

	x	*y*	*z*	B_{eq}
C1	−9 (4)	8262 (2)	−475 (5)	36 (1)
C2	−80 (5)	9444 (2)	−300 (5)	42 (1)
C3	1653 (6)	10067 (2)	1840 (5)	41 (1)
C4	3965 (5)	9889 (2)	1936 (5)	40 (1)
C5	4102 (4)	8719 (2)	1653 (4)	30 (1)
C6	5685 (5)	8359 (2)	3100 (4)	33 (1)
C7	5984 (5)	7226 (2)	2936 (4)	31 (1)
C8	4636	6563	633	24 (1)
C9	2259 (4)	6817 (2)	−99 (4)	25 (1)
C10	2318 (4)	8003 (2)	−379 (4)	27 (1)
C11	631 (4)	6033 (2)	−2168 (5)	33 (1)
C12	653 (4)	4858 (2)	−1988 (5)	31 (1)
C13	3031 (4)	4646 (2)	−1489 (4)	25 (1)
C14	4479 (4)	5379 (2)	724 (4)	25 (1)
C15	6650 (4)	4948 (2)	1448 (5)	33 (1)
C16	5966 (5)	3745 (2)	390 (5)	33 (1)
C17	3412 (4)	3556 (2)	−761 (4)	28 (1)
C18	3914 (5)	4816 (2)	−3456 (5)	35 (1)
C19	2857 (6)	8232 (2)	−2516 (5)	38 (1)
C20	2487 (5)	2612 (2)	−2652 (5)	37 (1)
C21	123 (6)	2048 (2)	−3069 (6)	46 (1)
C16*αM*	6690 (6)	3009 (3)	2065 (7)	52 (1)
O3*β*	1516 (5)	11164 (2)	1873 (5)	55 (1)
O20	3618 (5)	2330 (2)	−3751 (5)	59 (1)

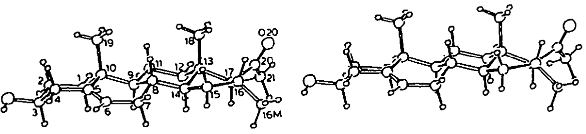

Fig. 1. *ORTEP*II (Johnson, 1976) stereoview of the molecule with atomic numbering.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C1—C2	1·524 (4)	C10—C19	1·541 (4)
C1—C10	1·554 (4)	C11—C12	1·534 (4)
C2—C3	1·513 (4)	C12—C13	1·526 (4)
C3—C4	1·509 (5)	C13—C14	1·539 (3)
C3—O3*β*	1·430 (4)	C13—C17	1·560 (4)
C4—C5	1·519 (4)	C13—C18	1·536 (4)
C5—C6	1·327 (4)	C14—C15	1·532 (4)
C5—C10	1·529 (3)	C15—C16	1·559 (4)
C6—C7	1·500 (4)	C16—C17	1·538 (4)
C7—C8	1·530	C16—C16*αM*	1·520 (5)
C8—C9	1·542	C17—C20	1·515 (3)
C8—C14	1·524	C20—C21	1.498 (4)
C9—C10	1·556 (3)	C20—O20	1·207 (5)
C9—C11	1·544 (3)		

C2—C1—C10	114·3 (2)	C9—C10—C19	112·0 (2)
C1—C2—C3	110·6 (2)	C9—C11—C12	113·9 (2)
C2—C3—C4	110·3 (2)	C11—C12—C13	110·8 (2)
C2—C3—O3*β*	107·5 (2)	C12—C13—C14	107·0 (2)
C4—C3—O3*β*	111·6 (2)	C12—C13—C17	116·7 (2)
C3—C4—C5	112·9 (2)	C12—C13—C18	110·8 (2)
C4—C5—C6	121·2 (2)	C14—C13—C17	98·4 (2)
C4—C5—C10	115·8 (2)	C14—C13—C18	113·2 (2)
C6—C5—C10	123·1 (2)	C17—C13—C18	110·3 (2)
C5—C6—C7	125·0 (2)	C8—C14—C13	114·2
C6—C7—C8	112·4	C8—C14—C15	118·8
C7—C8—C9	109·1	C13—C14—C15	104·8 (2)
C7—C8—C14	110·5	C14—C15—C16	105·1 (2)
C9—C8—C14	109·8	C15—C16—C17	104·6 (2)
C8—C9—C10	111·8	C15—C16—C16*αM*	113·6 (2)
C8—C9—C11	112·8	C17—C16—C16*αM*	113·9 (2)
C10—C9—C11	113·0 (2)	C13—C17—C16	103·4 (2)
C1—C10—C5	108·4 (2)	C13—C17—C20	114·4 (2)
C1—C10—C9	108·2 (2)	C16—C17—C20	114·3 (2)
C1—C10—C19	109·9 (2)	C17—C20—C21	116·7 (2)
C5—C10—C9	109·6 (2)	C17—C20—O20	121·1 (2)
C5—C10—C19	108·7 (2)	C21—C20—O20	122·2 (2)

C10—C1—C2—C3	−57·2 (3)	C11—C9—C10—C5	−175·4 (2)
C2—C1—C10—C5	50·2 (3)	C11—C9—C10—C19	−54·7 (3)
C2—C1—C10—C9	168·9 (2)	C8—C9—C11—C12	48·4
C2—C1—C10—C19	−68·5 (3)	C10—C9—C11—C12	176·4 (2)
C1—C2—C3—C4	57·2 (3)	C9—C11—C12—C13	−54·6 (3)
C1—C2—C3—O3*β*	179·1 (3)	C11—C12—C13—C14	58·6 (3)
C2—C3—C4—C5	−54·4 (3)	C11—C12—C13—C17	167·5 (2)
O3*β*—C3—C4—C5	−173·9 (3)	C11—C12—C13—C18	−65·2 (3)
C3—C4—C5—C6	−128·8 (3)	C12—C13—C14—C8	−61·9
C3—C4—C5—C10	51·2 (3)	C12—C13—C14—C15	166·3 (2)
C4—C5—C6—C7	−178·7 (3)	C17—C13—C14—C8	176·8
C10—C5—C6—C7	1·3 (4)	C17—C13—C14—C15	45·0 (2)
C4—C5—C10—C1	−46·9 (3)	C18—C13—C14—C8	60·4
C4—C5—C10—C9	−164·7 (2)	C18—C13—C14—C15	−71·4 (3)
C4—C5—C10—C19	72·6 (3)	C12—C13—C17—C16	−160·6 (2)
C6—C5—C10—C1	133·1 (3)	C12—C13—C17—C20	74·5 (3)
C6—C5—C10—C9	15·2 (4)	C14—C13—C17—C16	−46·7 (2)
C6—C5—C10—C19	−107·4 (3)	C14—C13—C17—C20	−171·6 (2)
C5—C6—C7—C8	13·8	C18—C13—C17—C16	71·8 (3)
C6—C7—C8—C9	−44·1	C18—C13—C17—C20	−53·1 (3)
C6—C7—C8—C14	−164·9	C8—C14—C15—C16	−155·8
C7—C8—C9—C10	62·7	C13—C14—C15—C16	−26·7 (3)
C7—C8—C9—C11	−168·6	C14—C15—C16—C17	−3.1 (3)
C14—C8—C9—C10	−176·0	C14—C15—C16—C16*αM*	−127·9 (3)
C14—C8—C9—C11	−47·4	C15—C16—C17—C13	31·3 (3)
C7—C8—C14—C13	176·6	C15—C16—C17—C20	156·3 (2)
C7—C8—C14—C15	−58·8	C16*αM*—C16—C17—C13	156·0 (3)
C9—C8—C14—C13	56·2	C16*αM*—C16—C17—C20	−79·0 (3)
C9—C8—C14—C15	−179·1	C13—C17—C20—C21	−90·8 (3)
C8—C9—C10—C1	−164·9	C13—C17—C20—O20	89·6 (3)
C8—C9—C10—C5	−46·9	C16—C17—C20—C21	150·3 (3)
C8—C9—C10—C19	73·8	C16—C17—C20—O20	−29·4 (4)
C11—C9—C10—C1	66·5 (3)		

tions with $I > 4\sigma(I)$. O3*β* and O20 form an intermolecular hydrogen bond; O···O = 2·82 Å, O—H···O = 164·3°, H···O = 2·08 Å. The C16—C17—C20—O20 torsion angle is in the normal range for the progesterone side chain.

Experimental. A wedge-shaped crystal was grown from chloroform. Crystal size 0·20 × 0·40 × 0·90 mm. Syntex *P*3 diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($20 < 2\theta < 35°$) checked with oscillation photographs, Mo *Kα* radiation, Nb-filtered, no monochromator, scan width variable, scan speed from 3 to 30° min^{-1} in 2θ, scan width $[2\cdot4 + 1\cdot04(2\theta_{K\alpha2} - 2\theta_{K\alpha1})]$, $2\theta_{\max} = 50°$, $-8 \le h \le 8$, $-18 \le k \le 18$, $-8 \le l \le 1$, 2832 independent reflections measured using a θ–2θ scan mode, 2587 with $I > 4\sigma(I)$. Five standard reflections ($\bar{1}8\bar{1}$, $1\bar{1}5$, 500, $24\bar{2}$, $\bar{2}4\bar{2}$) were measured every 100 reflections and varied in intensity by ≤5% during the data collection.

Direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least squares on *F* using the 2587 reflections for which $I > 4\sigma(I)$. The H positions were located in a difference

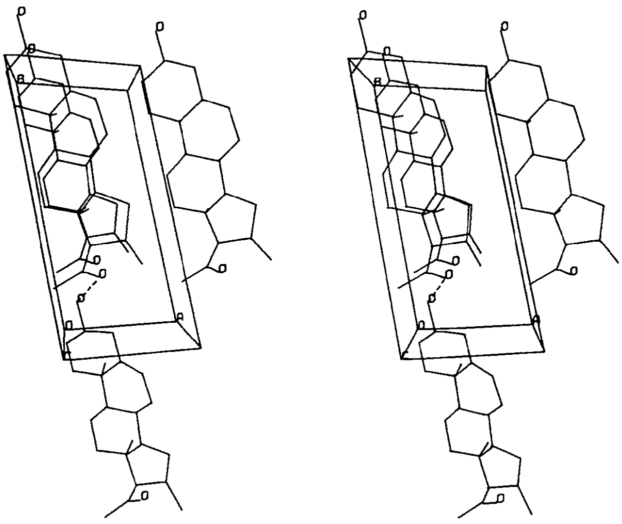

Fig. 2. Stereopacking of the molecule down the c axis, showing the hydrogen bonding.

map and refined with isotropic temperature parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}043$, $wR = 0{\cdot}050$, $S = 1{\cdot}210$ for observed reflections and $R = 0{\cdot}048$ for all data, $w = 1/\sigma^2$, $(\Delta/\sigma)_{\max} = 0{\cdot}19$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of $+0{\cdot}42$ and $-0{\cdot}15\ \text{e}\ \text{Å}^{-3}$. No corrections for absorption or extinction were made.

Atomic parameters are listed in Table 1. Distances, angles and selected torsion angles are listed in Table 2.* A stereoscopic view of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1. Fig. 2 shows a stereoview of the molecular packing.

Related literature. This structure is one of a series of steroid structures related to progesterone. Although 16β-substitution has been shown to destabilize the minimum energy conformation of the progesterone side chain, 16α-substitution does not have the same effect. The C16—C17—C20—O20 torsion angle, $-29{\cdot}4°$, is in the center of the observed range for 81 of 85 crystallographically observed pregnanes (Duax, Griffin & Rohrer, 1981).

---

* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51890 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Duax, W. L., Griffin, J. F. & Rohrer, D. C. (1981). *J. Am. Chem. Soc.* **103**, 6705–6712.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

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

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.

Acta Cryst. (1989). **C45**, 1435–1437

Structure of 7,10-Dimethoxy-2,6,6,9-tetramethyltricyclo[5.4.0.0^{1,5}]-undec-9-ene-8,11-dione*

By M. Soriano-García,† F. Walls, F. Yuste, R. Sánchez-Obregón, B. Ortíz, E. Díaz, R. A. Toscano and H. Barrios

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, *Mexico DF*

(*Received* 21 *February* 1989; *accepted* 4 *April* 1989)

Abstract. $C_{17}H_{24}O_4$, $M_r = 292{\cdot}4$, monoclinic, $P2_1$, $a = 7{\cdot}937\ (3)$, $b = 14{\cdot}031\ (6)$, $c = 8{\cdot}143\ (3)$ Å, $\beta = 116{\cdot}59\ (3)°$, $V = 811\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}20$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}078$ mm^{-1}, $F(000) = 316$, $T = 293$ K, $R = 0{\cdot}057$ for 780 observed reflections. The X-ray study confirms that in the solid state the structure of the title compound is similar to that inferred from chemical and spectroscopic evidence. The Cremer & Pople [*J. Am. Chem. Soc.* (1975), **97**, 1354–1358] ring-puckering parameters for the six-membered A ring and five-membered C ring are $\theta = 99\ (1)$, $\varphi = 120\ (1)°$, $Q = 0{\cdot}298\ (8)$ Å, and $\varphi = -119\ (1)°$, $Q =$

---

* Contribution No. 967 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.