$C_{23}H_{31}NO_2$



Fig. 1. View of (1) showing the atom-labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Methylene H atoms at C4 and C6, methyl H atoms at C7 and C9 and all phenyl ring H atoms have been omitted for clarity. Other H atoms are represented as spheres of arbitrary size.

**References**

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

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

GADOL, S. M. & DAVIS, R. E. (1982). *Organometallics*, **1**, 1607–1613.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1988). *SHELXTL-Plus.* Nicolet XRD, Madison, Wisconsin, USA.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

WHITESELL, J. K. & CARPENTER, J. (1987). *J. Am. Chem. Soc.* **109**, 2839–2840.

WHITESELL, J. K., CARPENTER, J., YASER, H. K. & MACHAJEWSKI, T. (1990). *J. Am. Chem. Soc.* **112.** Accepted for publication.

WHITESELL, J. K., CHEN, H.-H. & LAWRENCE, R. M. (1985). *J. Org. Chem.* **50**, 4663–4664.

WHITESELL, J. K. & LAWRENCE, R. M. (1986). *Chimia*, **40**, 318–321.

YASER, H. K. (1990). Masters thesis, Univ. of Texas at Austin, Austin, TX, USA.

# Structure of 6α-Chloro-3,20-dioxo-4-pregnen-17α-yl Acetate

By ZDZISLAW WAWRZAK AND PAWEL GROCHULSKI

*Technical University of Łodz, Institute of Physics, ul. Wolczanska 219, 93-005, Łodz, Poland*

ZDZISLAW GALDECKI

*Technical University of Łodz, Institute of General Chemistry, ul. Zwirki 36, 90-924 Łodz, Poland*

AND JANE F. GRIFFIN, PHYLLIS D. STRONG AND WILLIAM L. DUAX

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

**Abstract.** $C_{23}H_{31}ClO_4$, $M_r = 406.95$, orthorhombic, $P2_12_12_1$, $a = 13.155$ (2), $b = 13.397$ (2), $c = 12.369$ (2) Å, $V = 2179.9$ (9) Å$^3$, $Z = 4$, $D_x = 1.24$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.05$ cm$^{-1}$, $F(000) = 872$, $T = 293$ K, $R = 0.064$, $wR = 0.063$ for 2828 observed reflections with $F_o > 3\sigma(F_o)$. Rings *B* and *C* have chair conformations and the *D* ring is in an intermediate sofa–half-chair conformation. Ring *A* assumes an intermediate sofa–half-chair conformation and is flat relative to the rest of the steroid skeleton. The progesterone side chain has a conformation typical for other 17α-ester steroids; the C(16)—C(17)—C(20)—O(20) torsion angle is −23.0 (5)°.

**Experimental.** Crystal with dimensions $0.2 \times 1.2 \times 1.4$ mm, Nicolet *P3* diffractometer, cell dimensions and Laue symmetry from 25 centered reflections ($27 < 2\theta < 31°$) checked with oscillation photographs, Mo *Kα* radiation, Nb filtered, no monochromator, scan width $2.4° + 1.04$ ($2\theta_{K\alpha2} - 2\theta_{K\alpha1}$), scan speed from 3 to 30° min$^{-1}$ in $2\theta$, $2\theta_{\max} = 60°$, $0 \le h \le 19$, $0 \le k \le 19$, $0 \le l \le 18$, 4254 reflections were measured using $\theta$–$2\theta$ scan mode, 3588 of them were unique, $R_{int} = 0.026$. Four standard reflections (10,0,0; 080; 006; 565) were measured every 196 reflections and varied in intensity by $\le 5\%$ during the data collection. Intensity corrections made with the *DREAM* program (Blessing, 1987).

Direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-hydrogen atoms. The positional and anisotropic displacement parameters of all non-hydrogen atoms were refined by the full-

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic temperature parameters* $(\text{Å}^2 \times 10^3)$

| | $x$ | $y$ | $z$ | $U^*_{eq}$ |
|---|---|---|---|---|
| C(1) | 6402 (4) | 308 (4) | 7964 (4) | 69 (2) |
| C(2) | 7109 (5) | 808 (5) | 7164 (4) | 89 (2) |
| C(3) | 6704 (4) | 1790 (5) | 6808 (4) | 79 (2) |
| C(4) | 6104 (3) | 2329 (3) | 7584 (3) | 63 (1) |
| C(5) | 5873 (2) | 1998 (2) | 8585 (3) | 45 (1) |
| C(6) | 5396 (3) | 2631 (2) | 9437 (3) | 53 (1) |
| C(7) | 4498 (3) | 2143 (3) | 9974 (4) | 52 (1) |
| C(8) | 4785 (2) | 1126 (2) | 10437 (2) | 39 (1) |
| C(9) | 5214 (2) | 468 (2) | 9529 (3) | 39 (1) |
| C(10) | 6150 (2) | 943 (2) | 8952 (2) | 43 (1) |
| C(11) | 5408 (3) | −604 (3) | 9915 (4) | 55 (1) |
| C(12) | 4475 (3) | −1085 (3) | 10458 (4) | 56 (1) |
| C(13) | 4090 (2) | −434 (2) | 11389 (3) | 44 (1) |
| C(14) | 3864 (2) | 616 (2) | 10943 (3) | 41 (1) |
| C(15) | 3301 (3) | 1131 (3) | 11874 (4) | 59 (1) |
| C(16) | 2685 (3) | 286 (3) | 12398 (3) | 55 (1) |
| C(17) | 3026 (2) | −694 (3) | 11863 (3) | 47 (1) |
| C(18) | 4858 (3) | −416 (4) | 12326 (4) | 66 (1) |
| C(19) | 7080 (3) | 1014 (4) | 9710 (4) | 61 (1) |
| C(20) | 3048 (3) | −1606 (4) | 12614 (3) | 69 (1) |
| C(21) | 3068 (7) | −2627 (4) | 12134 (6) | 95 (2) |
| C(22) | 1435 (3) | −1215 (3) | 11114 (3) | 62 (1) |
| C(23) | 884 (4) | −1361 (5) | 10084 (5) | 83 (2) |
| Cl(6*A*) | 5013 (1) | 3844 (1) | 8967 (1) | 99 (1) |
| O(3) | 6911 (4) | 2130 (4) | 5931 (3) | 137 (2) |
| O(17*A*) | 2401 (2) | −915 (2) | 10932 (2) | 49 (1) |
| O(20) | 3123 (3) | −1497 (3) | 13579 (2) | 94 (1) |
| O(22) | 1108 (2) | −1334 (3) | 12001 (2) | 92 (1) |

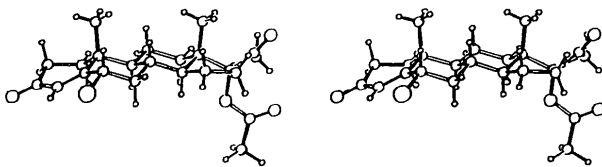$$U_{eq} = (1/3)\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$
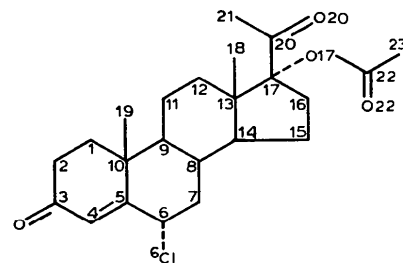


Fig. 1. Stereoview of the molecule.

matrix least-squares method on $F$ values using 2828 reflections for which $F_o > 3\sigma(F_o)$. The hydrogen-atom positions were located from a difference Fourier map and refined with isotropic displacement parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.064$ $(R_{all} = 0.090)$, $wR = 0.063$, $S = 2.157$ for 377 variables and 2828 reflections, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 1.26$ for H21*A* temperature parameter. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum negative and positive peaks of $-0.35$ and $0.33$ e $\text{Å}^{-3}$. No corrections for absorption or extinction were made.

Atomic positional and equivalent isotropic displacement parameters for non-hydrogen atoms are listed in Table 1. Bond lengths and valency angles

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

| | | | | |
|---|---|---|---|---|
| C(1)—C(2) | 1·514 (8) | C(11)—C(12) | 1·540 (6) |
| C(1)—C(10) | 1·525 (6) | C(12)—C(13) | 1·531 (5) |
| C(2)—C(3) | 1·486 (9) | C(13)—C(14) | 1·539 (4) |
| C(3)—C(4) | 1·437 (6) | C(13)—C(17) | 1·557 (4) |
| C(3)—O(3) | 1·207 (6) | C(13)—C(18) | 1·538 (6) |
| C(4)—C(5) | 1·350 (5) | C(14)—C(15) | 1·533 (5) |
| C(5)—C(6) | 1·491 (5) | C(15)—C(16) | 1·536 (6) |
| C(5)—C(10) | 1·529 (4) | C(16)—C(17) | 1·536 (5) |
| C(6)—C(7) | 1·505 (5) | C(17)—C(20) | 1·535 (6) |
| C(6)—Cl(6*A*) | 1·799 (3) | C(17)—O(17*A*) | 1·446 (4) |
| C(7)—C(8) | 1·525 (5) | C(20)—C(21) | 1·491 (7) |
| C(8)—C(9) | 1·535 (4) | C(20)—O(20) | 1·207 (5) |
| C(8)—C(14) | 1·525 (4) | C(22)—C(23) | 1·479 (7) |
| C(9)—C(10) | 1·560 (4) | C(22)—O(17*A*) | 1·352 (4) |
| C(9)—C(11) | 1·535 (4) | C(22)—O(22) | 1·188 (5) |
| C(10)—C(19) | 1·544 (5) | | |
| | | | |
| C(2)—C(1)—C(10) | 114·2 (4) | C(11)—C(12)—C(13) | 110·7 (3) |
| C(1)—C(2)—C(3) | 111·4 (4) | C(12)—C(13)—C(14) | 108·4 (2) |
| C(2)—C(3)—C(4) | 116·3 (4) | C(12)—C(13)—C(17) | 117·0 (3) |
| C(2)—C(3)—O(3) | 121·3 (4) | C(12)—C(13)—C(18) | 111·0 (3) |
| C(4)—C(3)—O(3) | 122·3 (4) | C(14)—C(13)—C(17) | 99·6 (2) |
| C(3)—C(4)—C(5) | 124·8 (3) | C(14)—C(13)—C(18) | 112·5 (3) |
| C(4)—C(5)—C(6) | 123·8 (3) | C(17)—C(13)—C(18) | 108·0 (3) |
| C(4)—C(5)—C(10) | 121·5 (3) | C(8)—C(14)—C(13) | 113·8 (2) |
| C(6)—C(5)—C(10) | 114·6 (2) | C(8)—C(14)—C(15) | 119·4 (3) |
| C(5)—C(6)—C(7) | 113·3 (3) | C(13)—C(14)—C(15) | 103·6 (2) |
| C(5)—C(6)—Cl(6*A*) | 113·7 (2) | C(14)—C(15)—C(16) | 103·9 (3) |
| C(7)—C(6)—Cl(6*A*) | 108·4 (3) | C(15)—C(16)—C(17) | 107·1 (3) |
| C(6)—C(7)—C(8) | 111·1 (3) | C(13)—C(17)—C(16) | 103·5 (2) |
| C(7)—C(8)—C(9) | 109·2 (2) | C(13)—C(17)—C(20) | 112·9 (3) |
| C(7)—C(8)—C(14) | 110·9 (2) | C(13)—C(17)—O(17*A*) | 104·9 (2) |
| C(9)—C(8)—C(14) | 109·6 (2) | C(16)—C(17)—C(20) | 115·2 (3) |
| C(8)—C(9)—C(10) | 113·0 (2) | C(16)—C(17)—O(17*A*) | 110·6 (2) |
| C(8)—C(9)—C(11) | 111·7 (2) | C(20)—C(17)—O(17*A*) | 109·3 (3) |
| C(10)—C(9)—C(11) | 113·1 (2) | C(17)—C(20)—C(21) | 119·3 (4) |
| C(1)—C(10)—C(5) | 109·3 (3) | C(17)—C(20)—O(20) | 120·2 (3) |
| C(1)—C(10)—C(9) | 108·1 (3) | C(21)—C(20)—O(20) | 120·2 (4) |
| C(1)—C(10)—C(19) | 110·4 (3) | C(23)—C(22)—O(17*A*) | 110·9 (3) |
| C(5)—C(10)—C(9) | 108·9 (2) | C(23)—C(22)—O(22) | 126·8 (3) |
| C(5)—C(10)—C(19) | 108·2 (3) | O(17*A*)—C(22)—O(22) | 122·3 (3) |
| C(9)—C(10)—C(19) | 111·9 (3) | C(17)—O(17*A*)—C(22) | 117·6 (2) |
| C(9)—C(11)—C(12) | 113·2 (3) | | |

are listed in Table 2.* The atomic numbering and molecular conformation are shown below and a stereoscopic view (Johnson, 1976) of the molecule is given in Fig. 1.



**Related literature.** A Cambridge Structural Database search (Allen, Kennard & Taylor, 1983) has revealed

---

* Lists of anisotropic displacement parameters, H-atom parameters, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53896 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

two 6$\alpha$-halogen-4-en-3-one steroid structures [BANDDO: 6$\alpha$-bromo-4-androstene-3,17-dione (Hazel, Strong, Duax & Osawa, 1977) and FTXPRO: 11$\beta$,17$\alpha$,21-trihydroxy-6$\alpha$-fluoro-4-preg-nene-3,20-dione (Duax & Norton, 1975)].

The overall geometry of the title molecule agrees well with the gemetry of the mean 4-en-3-one steroid molecule (Griffin, Duax & Weeks, 1984). The $B$ and $C$ rings have chair and the $D$ ring intermediate sofa–half-chair conformations with asymmetry parameters $\Delta C_s^{13} = 10\cdot2$ and $\Delta C_s^{16} = 10\cdot5$ (Duax & Norton, 1975). The $A$ ring has an intermediate sofa–half-chair conformation with asymmetry parameters $\Delta C_s^1 = 10\cdot9$ and $\Delta C_2^{1,2} = 15\cdot1$.

The C(16)—C(17)—C(20)—O(20) torsion angle has been observed to be confined to a narrow range −24 to −18° with the average value −21 (2)° in 17$\alpha$-ester steroids (Duax, Griffin & Rohrer, 1981). This torsion angle is −23·0 (5)° in the title molecule.

**References**

ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
BLESSING, R. H. (1987). *Crystallogr. Rev.* **1**, 3–58.
DUAX, W. L., GRIFFIN, J. F. & ROHRER, D. C. (1981). *J. Am. Chem. Soc.* **103**, 6705–6712.
DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1. New York: Plenum.
GRIFFIN, J. F., DUAX, W. L. & WEEKS, C. M. (1984). *Atlas of Steroid Structure*, Vol. 2. New York: Plenum.
HAZEL, J., STRONG, P., DUAX, W. L. & OSAWA, Y. (1977). *Cryst. Struct. Commun.* **6**, 507–510.
JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
KIM, R. S., LABELLA, F. S., ZUNZA, H., ZUNZA, F. & TEMPLETON, J. F. (1980). *Mol. Pharmacol.* pp. 395–398.
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.

---

# Structure du 2,3-Diméthyl-3-(1,2-diméthylimidazol-4-yl)butan-2-ol*

PAR MICHEL P. CROZET

*URA CNRS 'Radicaux Libres et Synthèse', Faculté des Sciences et Techniques de St Jérôme, Avenue Escadrille Normandie–Niemen, 13397 Marseille CEDEX 13, France*

PATRICE VANELLE

*Laboratoire de Chimie Organique, Faculté de Pharmacie, 27 Boulevard Jean Moulin, 13385 Marseille CEDEX 5, France*

ET ANDRÉ BALDY ET FOUAD RIDOUANE

*Laboratoire de Chimie Inorganique Moléculaire, Associé au CNRS, Faculté des Sciences et Techniques de St Jérôme, Avenue Escadrille Normandie–Niemen, 13397 Marseille CEDEX 13, France*

**Abstract.** 2,3-Dimethyl-3-(1,2-dimethylimidazol-4-yl)-butan-2-ol, $C_{11}H_{20}N_2O$, monoclinic, $P2_1/c$, $M_r = 196\cdot29$, $a = 12\cdot53$ (4), $b = 7\cdot38$ (2), $c = 13\cdot09$ (5) Å, $\beta = 95\cdot01$ (1)°, $V = 1205\cdot8$ (2) Å$^3$, $Z = 4$, $F(000) = 432$, $D_x = 1\cdot08$ g cm$^{-3}$, Mo $K\alpha = 0\cdot71069$ Å, $\mu = 7\cdot7$ cm$^{-1}$, $T = 298$ K, $R = 0\cdot065$ for 1395 reflections with $I > 3\sigma$. The structure of the tertiary alcohol formed in the first example of hydrolysis of a tertiary nitroalkane is shown to be 2,3-dimethyl-3-(1,2-dimethylimidazol-4-yl)butan-2-ol.

**Partie expérimentale.** Les 5-nitroimidazoles représentent une famille de médicaments d'une grande importance dans le traitement de l'amibiase,