Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 3β-Acetoxy-7α-chloro-5,6β-epoxy-15β,16β-methylene-5β-androstan-17-one

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Received 2 December 2006
Accepted 22 December 2006

**Key indicators**

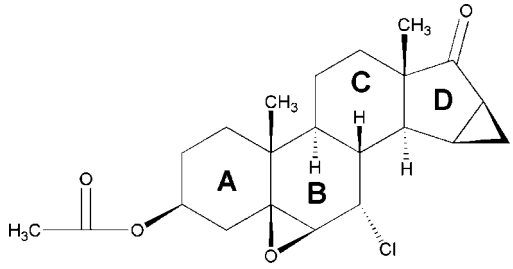
Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.053
*wR* factor = 0.128
Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title molecule, $C_{22}H_{29}ClO_4$, all bond lengths and angles show normal values. Rings *A* and *C* have slightly distorted chair conformations, while rings *B* and *D* show envelope conformations. Weak intermolecular C—H···O hydrogen bonds link the molecules into chains running along the *b* axis.

## Comment

Drospirenone is a new contraceptive drug with special anti-mineralocorticoid and anti-androgenic properties (Muhn *et al.*, 1995). In our attempts to synthesize drospirenone, the title compound, (I), was obtained as an intermediate *via* chlorination with triphenylphosphine and carbon tetrachloride from the corresponding precursor, 3β-acetoxy-5,6β-epoxy-7β-hydroxy-15β,16β-methylene-5β-androstan-17-one, (II). We report here the crystal structure of (I).




(I)

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987). In the cyclopropyl ring C15/C16/C20, the bond lengths C16—C20 and C15—C20 are different, at 1.511 (5) and 1.473 (4) Å, respectively, while the same bond lengths in the analogous structure of 3β-acetoxy-17,17-ethylenedioxy-15β,16β-methylene-5-androsten-7β-ol (Zhou & Hu, 2006) are almost equal [1.499 (6) and 1.493 (6) Å, respectively]. The difference in these bond lengths in (I) is most probably caused by a hyperconjugation interaction between the C=O π-bonding and C16—C20 σ-bonding orbitals.

Rings *A* and *C* show slightly distorted chair conformations, while rings *B* and *D* demonstrate envelope conformations. In ring *B*, atom C8 deviates by 0.652 (3) Å from the mean plane of atoms C5–C7/C9/C10, which makes a dihedral angle of 77.8 (1)° with the epoxy ring C5/C6/O3. In ring *D*, atom C13 deviates by 0.539 (7) Å from the mean plane of atoms C14–C17, which makes a dihedral angle of 63.3 (2)° with the cyclopropyl ring C15/C16/O20.

In the crystal structure of (I) (Fig. 2), weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into one-dimensional chains running along the *b* axis.

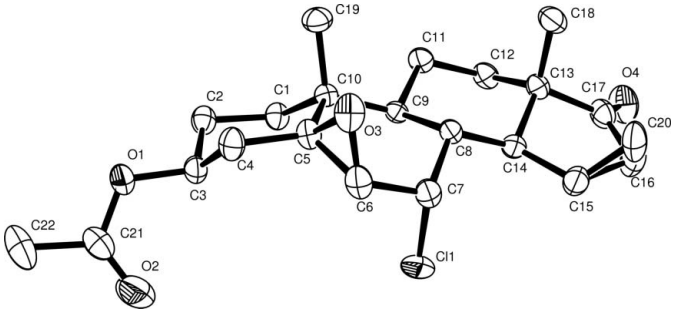



**Figure 1**
The molecular structure of (I), with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

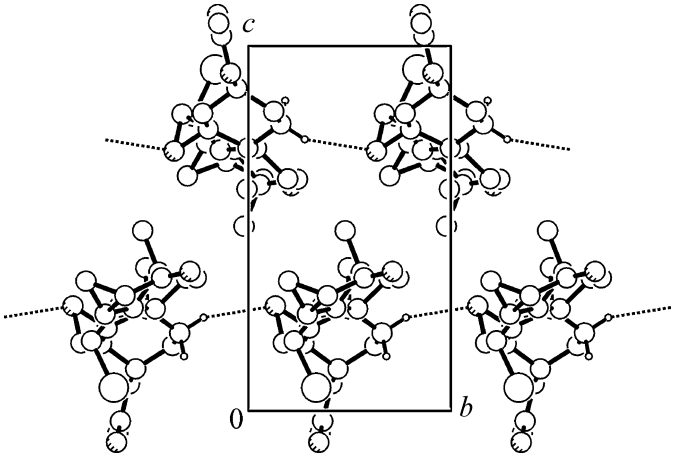



**Figure 2**
A portion of the crystal packing of (I), viewed down the *a* axis. Dashed lines denote weak C—H···O hydrogen bonds. Only two H atoms, H1*A* and H1*B*, are drawn for clarity.

## Experimental

Compound (II) was kindly donated by Mr Pan, Jiubang Chemistry Corporation Ltd., Shanghai, China. The title compound was synthesized from (II) *via* chlorination with triphenylphosphine and carbon tetrachloride, according to the literature method (Bittler *et al.*, 1984). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from isopropyl ether.

*Crystal data*

$C_{22}H_{29}ClO_4$
$M_r$ = 392.90
Monoclinic, $P2_1$
$a$ = 11.741 (5) Å
$b$ = 6.911 (3) Å
$c$ = 12.457 (5) Å
$\beta$ = 90.958 (5)°
$V$ = 1010.7 (7) Å$^3$
$Z$ = 2
$D_x$ = 1.291 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.21 mm$^{-1}$
$T$ = 293 (2) K
Prism, colourless
0.20 × 0.15 × 0.14 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: none
4915 measured reflections
4150 independent reflections
3484 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 27.0°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.128
$S$ = 0.98
4150 reflections
248 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.062 (5)
Absolute structure: Flack (1983), with 2020 Friedel pairs
Flack parameter: −0.01 (7)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1$A$···O3$^{i}$ | 0.97 | 2.59 | 3.480 (3) | 153 |

Symmetry code: (i) $x, y+1, z$.

All H atoms were placed in calculated positions, with C—H = 0.96–0.98 Å, and refined as riding, with $U_{iso}$(H) = 1.2–1.5$U_{eq}$ of the parent atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors are indebted to the Science and Technology Bureau of Zhejiang Province for financial surport (grant No. 2005C23022) and to Mr Pan for the kind donation of the precursor compound.

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bittler, D., Laurent, H., Nickisch, K., Nickolson, R. & Wiechert, R. (1984). US Patent 4 472 310.
Bruker (1997). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Muhn, P., Fuhrmann, U., Fritzemeier, K. H., Krattenmacher, R. & Schillinger, E. (1995). *Ann. N. Y. Acad. Sci.* **761**, 311–335.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhou, W., Zhong, G.-X., Hu, W.-X. & Xia, C.-N. (2006). *Acta Cryst.* E**62**, o3542–3543.