

Wei Zhou, Guang-Xiang Zhong,  
Wei-Xiao Hu\* and  
Chun-Nian XiaCollege of Pharmaceutical Science, Zhejiang  
University of Technology, Hangzhou 310014,  
People's Republic of China

Correspondence e-mail: huyang@mail.hz.zj.cn

## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.044  
 $wR$  factor = 0.120  
Data-to-parameter ratio = 8.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**3 $\beta$ -Acetoxy-17,17-ethylenedioxy-15 $\beta$ ,16 $\beta$ -  
methylene-5-androsten-7 $\beta$ -ol**

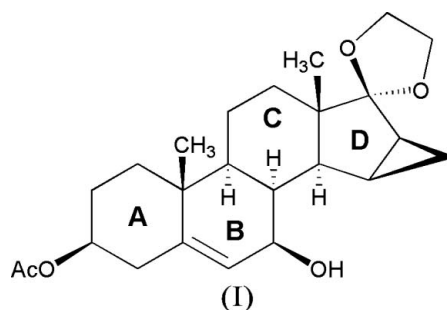
In the title compound,  $\text{C}_{24}\text{H}_{34}\text{O}_5$ , the cyclohexane rings adopt chair conformations while the cyclohexene ring is in a half-chair conformation. Both five-membered rings adopt envelope conformations.

Received 10 July 2006

Accepted 21 July 2006

## Comment

Drospirenone is a novel progestogen with antimineralecorticoid and anti-androgenic activity. In our attempt to synthesize drospirenone, the title compound, (I), was obtained as an intermediate from the corresponding ketone by stereoselective reduction with lithium tri-*tert*-butoxyaluminumhydride. We report here the crystal structure of (I).



The molecular structure of (I) is illustrated in Fig. 1. The  $\text{Csp}^3-\text{Csp}^3$  bond lengths in the steroid nucleus lie in the range 1.492 (6)–1.559 (5) Å. The  $\text{C5}=\text{C6}$  distance of 1.323 (5) Å is indicative of a double bond. All these distances are in close agreement with those in similar steroid structures (Rende & Trotter, 1974; Grochulski & Wawrzak, 1989).

As seen in Fig. 1, rings A and C adopt chair conformations. Ring B adopts a half-chair conformation, with atoms C8 and C9 deviating from the C5–C7/C10 plane by 0.309 (8) and  $-0.439$  (8) Å, respectively. Ring D is in an envelope conformation; the deviation of atom C13 from the C14–C17 plane is 0.567 (6) Å. The three-membered ring (C15/C1/C22) makes an angle of 63.3 (3)° with the C14–C17 plane. The dioxalane ring adopts an envelope conformation, with atom C23 at the flap position. The dihedral angle between the C14–C17 and O4/O5/C17/C24 planes is 79.9 (2)°.

Except for two weak intramolecular C–H...O interactions (Table 1), no other hydrogen bonds are found in the crystal structure of (I).

## Experimental

3 $\beta$ -Acetoxy-17,17-ethylenedioxy-15 $\beta$ ,16 $\beta$ -methylene-5-androsten-7-one was obtained from Mr Pan, Jiubang Chemistry Corporation Ltd.,

Shanghai, China. Compound (I) was synthesized according to a literature method by reducing the above ketone with lithium tri-*tert*-butoxyaluminumhydride (Bittler *et al.*, 1984). Single crystals of (I) suitable for X-ray diffraction study were obtained by slow evaporation of an acetone solution.

#### Crystal data

$C_{24}H_{34}O_5$	$Z = 4$
$M_r = 402.51$	$D_x = 1.269 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 10.049 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 11.550 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 18.149 (7) \text{ \AA}$	Prism, colourless
$V = 2106.5 (11) \text{ \AA}^3$	$0.50 \times 0.40 \times 0.35 \text{ mm}$

#### Data collection

Enraf–Nonius CAD-4 diffractometer	1415 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{\text{int}} = 0.016$
Absorption correction: none	$\theta_{\text{max}} = 25.2^\circ$
2307 measured reflections	3 standard reflections
2165 independent reflections	frequency: 60 min
	intensity decay: none

#### Refinement

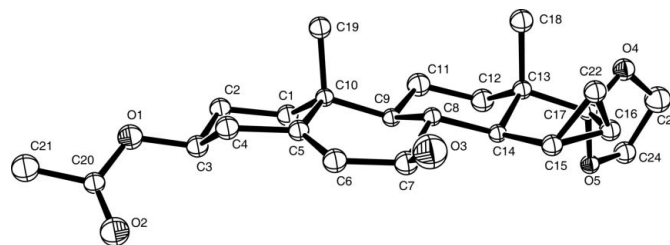
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.9275P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.12$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2165 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
267 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	(Sheldrick, 1997)
	Extinction coefficient: 0.0097 (11)

**Table 1**

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C12-H12A\cdots O5$	0.97	2.51	2.857 (5)	101
$C18-H18A\cdots O4$	0.96	2.38	2.774 (6)	104

The hydroxyl H atom was found in a difference Fourier map and refined with an O–H distance restraint of 0.82 (2)  $\text{\AA}$ . The other H atoms were placed in calculated positions and refined using a riding



**Figure 1**

The structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted.

model, with  $C-H = 0.96-0.98 \text{ \AA}$  and  $U_{\text{iso}}(H) = 1.2$  (or 1.5 for methyl H) times  $U_{\text{eq}}(C)$ . In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement. The absolute stereochemistry of the title compound was known from the synthetic route (Bernstein *et al.*, 1957).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4*, *PSI* and *EAC* in *CAD-4 EXPRESS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Science and Technology Bureau of Zhejiang Province for financial support (grant No. 2005 C23022). The authors express deep appreciation to Mr Pan for generously providing the sample.

#### References

- Bernstein, S., Stolar, S. M. & Heller, M. (1957). *J. Org. Chem.* **22**, 472–473.  
 Bittler, D., Laurent, H., Nickisch, K., Nickolson, R. & Wiechert, R. (1984). US Patent No. 4472310.  
 Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Grochulski, P. & Wawrzak, Z. (1989). *J. Crystallogr. Spectrosc. Res.* **19**, 577–587.  
 Rende, D. F. & Trotter, J. (1974). *J. Chem. Soc. Perkin Trans. 2*, pp. 847–852.  
 Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.