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Methyl 17,17-ethylenedioxy-7-oxo-15β,16β-methylene-5-androstene-3β-carboxylate

Wei Zhou, Wei-Xiao Hu* and Chun-Nian Xia

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

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

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Key indicators

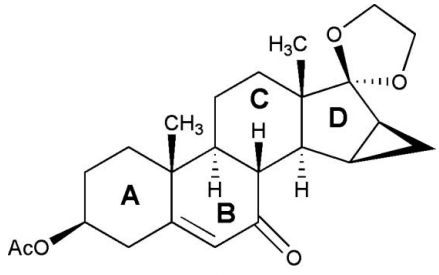
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.154
Data-to-parameter ratio = 8.2

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

The title compound, $C_{24}H_{32}O_5$, has rings *A* and *C* in regular chair, ring *B* in a distorted half-chair and ring *D* in an envelope conformation. The dioxolane ring adopts a twist conformation.

Comment

Drospirenone is a new contraceptive drug with special anti-mineralocorticoid and anti-androgenic properties, unlike any other progestin available in oral contraceptives. In our procedure to synthesize drospirenone, the title compound, (I), was obtained as an intermediate by oxidation with chromium trioxide and 3,5-dimethylpyrazole from the corresponding precursor, methyl 17,17-ethylenedioxy-15β,16β-methylene-5-androstene-3β-carboxylate.



(I)

The molecular structure of (I) is illustrated in Fig. 1 and a packing diagram is shown in Fig. 2. In the molecule, the Csp^3—Csp^3 bond lengths in the steroid nucleus show quite a scatter, ranging from 1.488 (7) to 1.558 (6) Å.

Rings *A* and *C* show a regular chair, ring *B* a distorted half-chair and ring *D* an envelope conformation; the deviation of atom C13 from the plane of atoms C14–C17 is 0.539 (7) Å. The three-membered ring (C15/C16/C22) makes a dihedral angle of 63.3 (3)° with the C14–C17 plane. The dioxolane ring adopts a twist conformation.

As shown in Fig. 2, there exist intramolecular hydrogen bonds and an intermolecular hydrogen bond in the crystal structure (Table 1).

Experimental

Methyl 17,17-ethylenedioxy-15β,16β-methylene-5-androstene-3β-carboxylate was obtained from Mr Pan, Jiubang Chemistry Corporation Ltd, Shanghai, China. The title compound was synthesized according to the literature by oxidizing the above compound with chromium trioxide and 3,5-dimethylpyrazole (Bittler et al., 1984). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an acetone solution.

Crystal data

$C_{24}H_{32}O_5$
M_r = 400.50
Orthorhombic, $P2_12_12_1$
a = 6.1020 (17) Å
b = 14.450 (3) Å
c = 23.763 (8) Å
V = 2095.3 (10) Å^3
Z = 4
D_x = 1.270 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Plate, colourless
0.50 × 0.40 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2301 measured reflections
2190 independent reflections
1285 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.2°
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.154
S = 1.06
2190 reflections
266 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.3809P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.006 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2	0.98	2.26	2.635 (8)	101
C15—H15···O3	0.98	2.54	2.949 (7)	105
C23—H23A···O3^{i}	0.97	2.40	3.333 (8)	162

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

The absolute stereochemistry of the title compound was known from the synthetic route (Bernstein et al., 1957). H atoms were placed at calculated positions and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C) and C—H = 0.96–0.98 Å. In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; 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 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

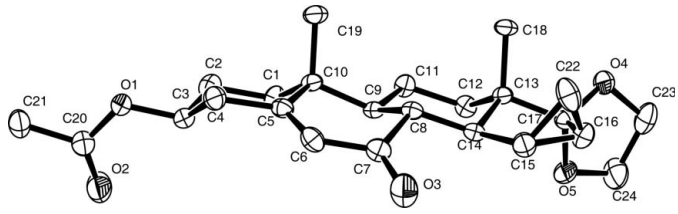


Figure 1
The structure of (I) with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

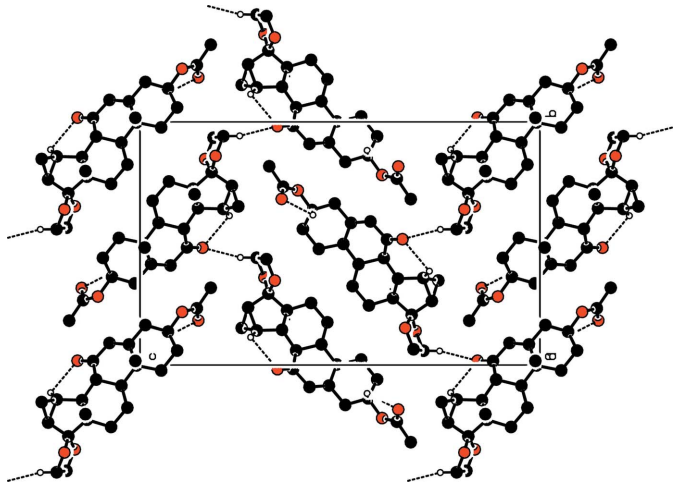


Figure 2
Packing diagram of (I), viewed along the a axis, showing hydrogen bonds as dashed lines. For clarity, H atoms not involved in hydrogen bonding have been omitted.

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