Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

20-Oxopregna-5,16-dien-3β-yl acetate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.143
Data-to-parameter ratio = 6.9

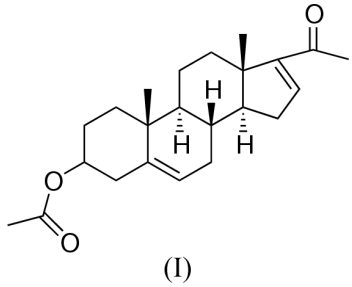
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{23}H_{32}O_3$, there are four fused rings, three six-membered and one five-membered. The two cyclohexane rings adopt chair conformations and the cyclohexene ring displays an envelope conformation.

Received 2 August 2004
Accepted 16 August 2004
Online 28 August 2004

Comment

The title compound, (I), is a key intermediate in the synthesis of progesterone, an important steroid hormone (Robert & Marinus, 2000). The molecule contains four fused rings, three six-membered and one five-membered (Fig. 1). The two six-membered rings (C3–C8 and C11–C16) adopt chair conformations, while the other six-membered ring (C5/C6/C12/C11/C10/C9) displays an envelope conformation. In the five-membered ring, atoms C14/C19/C18/C17 are nearly coplanar and atom C13 deviates from the plane by 0.577 (5) Å, also indicating an envelope conformation. Due to steric effects, some bond lengths and angles deviate from normal values (Table 1). The C18—C19—C20—O3 torsion angle of −172.9 (4)° clearly shows that the C20═O3 carbonyl group and C18═C19 double bond are approximately coplanar, with a common plane with an *S-trans* configuration.



(I)

Experimental

The title compound, (I), was a gift from Zhejiang XianJun Pharmaceutical Co. Ltd. It was recrystallized from ethanol, giving crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_{23}H_{32}O_3$
M_r = 356.50
Orthorhombic, $P2_12_12_1$
a = 8.8184 (4) Å
b = 12.5785 (5) Å
c = 18.6036 (2) Å
V = 2063.6 (1) Å^3
Z = 4
D_x = 1.147 Mg m^{-3}
Mo Kα radiation
Cell parameters from 14 681 reflections
θ = 1.1–27.5°
μ = 0.07 mm^{-1}
T = 296 (1) K
Plate, colorless
0.60 × 0.52 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.946, T_{max} = 0.993
18 683 measured reflections
2691 independent reflections
1615 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.044
θ_{max} = 27.5°
$h = -10 \rightarrow 11$
$k = -16 \rightarrow 16$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.143
S = 1.01
1615 reflections
235 parameters
H-atom parameters constrained
$w = 1/[0.0038F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—C7	1.557 (4)	C11—C12	1.540 (4)
C6—C12	1.551 (4)	C12—C16	1.541 (4)
C6—C22	1.539 (4)	C13—C14	1.542 (4)
C5—C9—C10	124.6 (3)	C13—C14—C19	99.6 (2)
C6—C7—C8	115.2 (3)	C17—C13—C14	103.7 (2)
C6—C12—C16	114.3 (2)	C13—C17—C18	100.4 (2)
C11—C13—C14	114.8 (2)	C14—C19—C20	124.6 (3)
C12—C16—C15	115.2 (2)	C18—C19—C20	124.8 (3)

In the absence of significant anomalous dispersion effects, Friedel-pair reflections were merged before the final refinement. The absolute configuration was assigned on the basis of the structure of related compounds having the analogous steroid skeleton (Bohl *et al.*, 1985; Broess *et al.*, 1997). H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the refinement in the riding model approximation, with U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

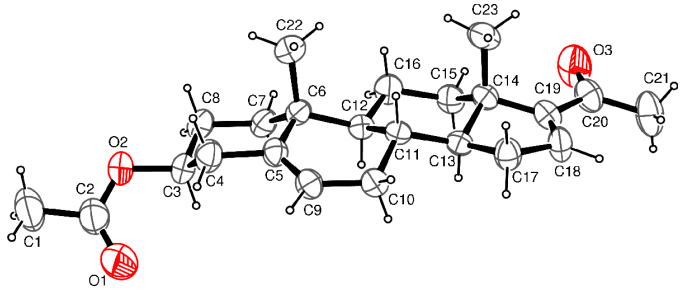


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii.

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