Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Pregna-1,4,9(11),16-tetraene-3,20-dione

Guo-Lin Zhang[a]* and Hong-Yun Peng[b]

[a]Department of Medicinal Chemistry, College of Pharmaceutical Science, Zhejiang University, People's Republic of China, and [b]MOE Key Laboratory of Environmental Remediation and Ecosystem Health, College of Natural Resources and Environmental Science, Zhejiang University, People's Republic of China

Correspondence e-mail: guolinzhang@zju.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.124
Data-to-parameter ratio = 10.7

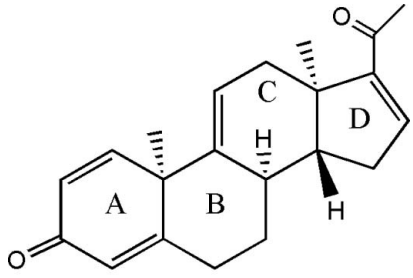
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{21}H_{24}O_2$, contains four rings. The cyclohexadienone ring is planar, the cyclohexane and cyclohexene rings adopt chair and sofa conformations, respectively, while the cyclopentene ring adopts an envelope conformation. Weak intermolecular C—H···O hydrogen bonding helps to stabilize the crystal structure.

Received 21 September 2006
Accepted 5 November 2006

Comment

Pregna-1,4,9(11),16-tetraene-3,20-dione derivatives are intermediates in the synthesis of steroid agents (Conrow, 1999; Boivin *et al.*, 1992; Rondinone *et al.*, 1992). The title compound, (I), is an intermediate in the synthesis of 21-chloro steroids (Annen *et al.*, 1982; Wuts *et al.*, 1993). The structure determination of (I) was carried out in order to determine the molecular conformation.



(I)

The molecular structure of (I) is shown in Fig. 1. The cyclohexadienone ring is planar. The cyclohexane ring adopts a chair conformation, the cyclohexene ring adopts a sofa conformation and the cyclopentane ring adopts an envelope conformation. Intermolecular weak C—H···O hydrogen bonding is observed in the crystal structure (Table 1).

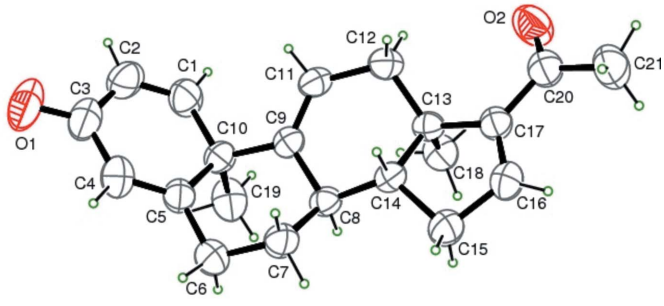


Figure 1
The molecular structure of (I), shown with 40% probability displacement ellipsoids (arbitrary spheres for H atoms).

Experimental

Compound (I) was supplied by Yier Weizhiyang Pharmaceutical Co. Ltd, China. Single crystals were obtained by slow crystallization from methanol–dichloromethane (1:1 *v*/*v*) at room temperature.

Crystal data

$C_{21}H_{24}O_2$
M_r = 308.40
Orthorhombic, $P2_12_12_1$
a = 6.6433 (19) Å
b = 11.278 (4) Å
c = 22.951 (8) Å
V = 1719.6 (10) Å^3
Z = 4
D_x = 1.191 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
Chunk, colorless
0.42 × 0.36 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
17000 measured reflections
2267 independent reflections
1393 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.124
S = 1.11
2267 reflections
212 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.1715P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.026 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C19—H19*C*···O1^{i}	0.96	2.55	3.505 (4)	173
C21—H21*B*···O2^{ii}	0.96	2.58	3.524 (5)	170

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

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined [U_{iso}(H) = 1.5U_{eq}(C)]. Other H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was not determined.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia,1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are grateful to Mr Jian-Ming Gu in the Analysis Center of Zhejiang University for providing analysis and helpful discussions.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Annen, K., Hofmeister, H., Laurent, H. & Wiechert, R. (1982). *Liebigs Ann. Chem.* **5**, 966–972.
Boivin, J., Chauvet, C. & Zard, Z. (1992). *Tetrahedron Lett.* **34**, 4913–4916.
Conrow, E. (1999). *J. Org. Chem.* **3**, 1042–1044.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.00. Rigaku/MSC, The Woodlands, Texas, USA.
Rondinone, C. M., Schillaci, R. & Roldan, A. (1992). *Drug. Dev. Res.* **1**, 61–65.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Wuts, P. G. M., Cabaj, J. E. & Maisto, K. D. (1993). *Synth. Commun.* **15**, 2199–2211.