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3a,5b-Dimethyl-2,3,3a,4,5,5a,5b,6,7,13,13a,13b-dodecahydro-1*H*-8-azacyclopenta[*a*]chrysen-3-ol

Ji-Zhong Yan, Jian Li and Guo-Wu Rao*

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

Correspondence e-mail: rgw@zjut.edu.cn

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Key indicators

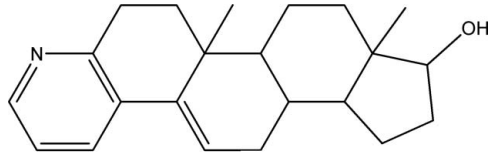
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.112
Data-to-parameter ratio = 10.7

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

The title compound, $C_{22}H_{29}NO$, is built up from five fused rings, four of which are six-membered and one five-membered. The ring adjacent to the five-membered ring adopts a chair conformation.

Comment

Testosterone derivatives have a high biological activity and have been widely used in the preparation of hormone-based drugs (Alvarez-Ginarte *et al.*, 2005). As part of our continuing interest in the structure–activity relationship of testosterone derivatives, we have isolated the product, (I), from the reaction of propargylamine and 17-hydroxy-10,13-dimethyl-1,7,8,10,11,12,13,15,16,17-decahydro-2*H*-cyclopenta[*a*]phenanthren-3(4*H*,9*H*,14*H*)-one.



(I)

The molecular structure of (I) is illustrated in Fig. 1. It is built up from five fused rings, four of which are six-membered and one five-membered. The C4/C5/C17–C20 ring fused with the five-membered ring adopts a chair conformation (Cremer & Pople, 1975). The other rings have no obvious conformations. There is an O—H···N hydrogen-bond interaction, building a zigzag chain parallel to the *a* axis (Table 1 and Fig. 2).

Experimental

The title compound was prepared according to the procedure of Yan *et al.* (2006). Crystals were obtained by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_{22}H_{29}NO$
M_r = 323.46
Orthorhombic, $P2_12_12_1$
a = 8.629 (2) Å
b = 13.664 (5) Å
c = 15.158 (4) Å
V = 1787.2 (9) Å^3
Z = 4
D_x = 1.202 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
Chunk, colorless
0.39 × 0.28 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
17581 measured reflections
2326 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°

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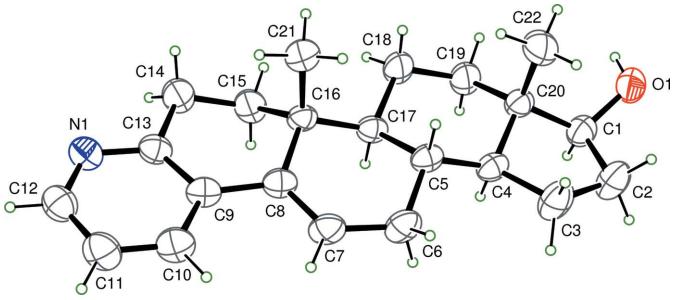

Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 0.0296P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.113$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
2326 reflections	Extinction correction: *SHELXL97*
218 parameters	Extinction coefficient: 0.0045 (18)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.82	2.04	2.862 (2)	178

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

H atoms were included in calculated positions and refined using a riding model, with U_{iso}(H) = 1.2 (or 1.5 for methyl H atoms) times U_{eq}(C). C—H distances were constrained to 0.96 Å for methyl H atoms, 0.97 Å for methylene H atoms, 0.98 Å for methine H atoms, and 0.93 Å for the remainder, and O—H = 0.82 Å. In the absence of significant anomalous scattering, Friedel pairs were averaged, and the absolute configuration assigned arbitrarily.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); 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: *WinGX* (Farrugia, 1999).

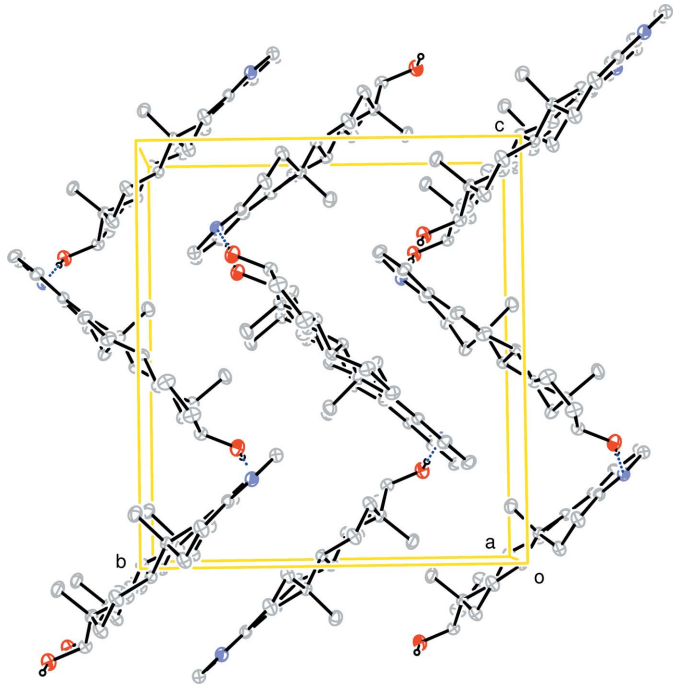

Figure 2
View showing the O—H···N hydrogen bonding (dashed lines) and the formation of the zigzag chain. For clarity, only H atoms involved in hydrogen bonding are shown. [Symmetry code: (i) $-x+\frac{1}{2}, -y+1, +z+\frac{1}{2}$.]

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References

Alvarez-Ginarte, Y. M., Crespo, R., Montero-Cabrera, L. A., Ruiz-Garcia, J. A., Ponce, Y. M., Santana, R., Pardillo-Fontdevila, E. & Alonso-Becerra, E. (2005). *QSAR Combin. Sci.* **24**, 218–226.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
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 3.6.0. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Yan, J.-Z., Li, J. & Rao, G.-W. (2006). *Acta Cryst.* E**62**, o2773–o2774.