Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

11,11a,13a-Trimethyl-2,3,3a,3b,4,5,5a,6,11,11a,-11b,12,13,13a-tetradecahydro-1*H*-7-azaindeno-[5,4-*a*]anthracen-1-ol methanol solvate

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

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.119
Data-to-parameter ratio = 11.3

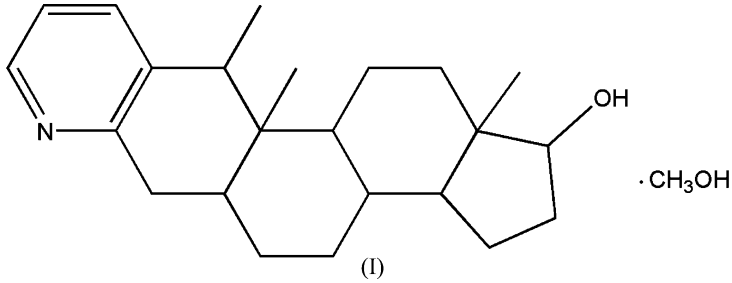
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{23}H_{33}NO\cdot CH_4O$, the asymmetric unit consists of one molecule of $C_{23}H_{33}NO$ and one methanol solvent molecule, which are connected through O—H···N and O—H···O hydrogen bonds leading to the formation of zigzag-like chains parallel to the *a* axis.

Received 23 May 2006
Accepted 5 June 2006

Comment

Testosterone derivatives exhibit a high level of biological activity and have been widely used as hormone treatments (Alvarez-Ginarte *et al.*, 2005). As part of our continuing interest in the structure–activity relationship of testosterone derivatives, we report here the structure of the title compound.



(I)

The asymmetric unit of the title compound consists of one molecule of $C_{23}H_{33}NO$ and one methanol solvent molecule, which are connected through O—H···N hydrogen bond (Table 1 and Fig. 1). The occurrence of a second hydrogen-

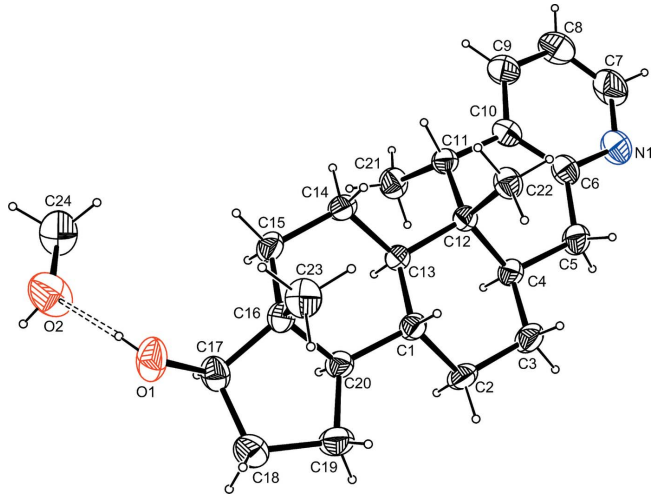


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii. The hydrogen bond is shown as dashed lines.

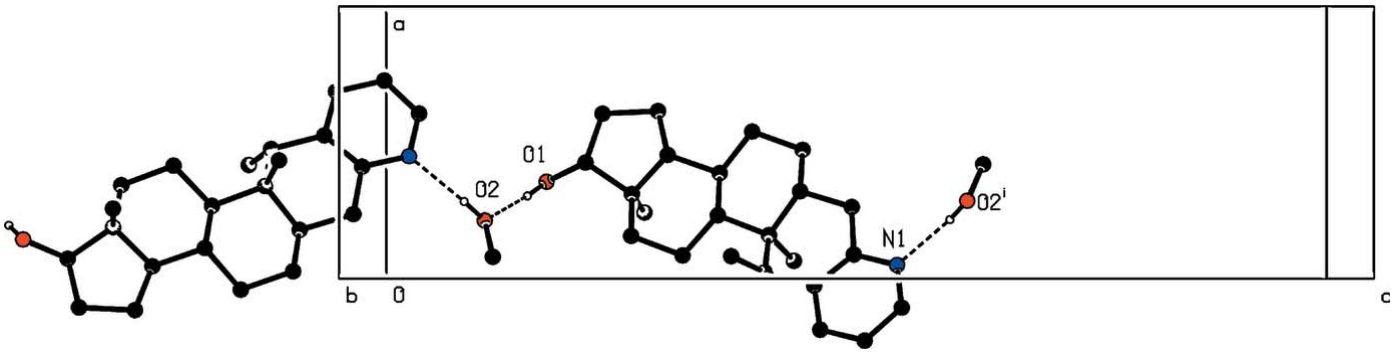


Figure 2
View showing the O—H···N and O—H···O hydrogen bonding and the formation of the zigzag-like chain. The hydrogen bonds are shown as dashed lines and H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$].

bonding interaction, O—H···O, between the H atom of the hydroxyl group of the molecule and the O atom of the solvent molecule, results in the formation of a zigzag-like 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) by reaction of propargylamine and 17-hydroxy-1,10,13-trimethyldodecahydro-2*H*-cyclopenta[*a*]phenanthren-3(4*H*,9*H*,14*H*)-one. A solution in methanol was gradually concentrated at room temperature to afford colourless crystals suitable for X-ray analysis.

Crystal data

$C_{23}H_{33}NO\cdot CH_4O$
$M_r = 371.55$
Orthorhombic, $P2_12_12_1$
$a = 7.4389\ (19)$ Å
$b = 10.599\ (4)$ Å
$c = 27.299\ (8)$ Å
$V = 2152.4\ (12)$ Å^3
$Z = 4$
$D_x = 1.147$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 298.1$ K
Chunk, colourless
0.46 × 0.42 × 0.33 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (Higashi, 1995)
$T_{min} = 0.957$, $T_{max} = 0.966$
4930 measured reflections
2825 independent reflections
2283 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.119$
$S = 1.04$
2825 reflections
251 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.4327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.012 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	1.94	2.750 (3)	170
O2—H2···N1^{i}	0.82	1.96	2.746 (3)	161

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

H atoms were included in calculated positions and treated as riding on their parent atoms with C—H distances of 0.96 Å (CH_3), 0.97 Å (CH_2), 0.98 Å ($CH_{methine}$) and 0.93 Å ($CH_{aromatic}$), O—H distances of 0.82 Å, and $U_{iso} = 1.5U_{eq}$(C,O) for CH_3 and hydroxy H atoms and $1.2U_{eq}$(C) for all others. As the compound contains no atoms heavier than Si, the absolute configuration could not be determined unambiguously and has been assigned arbitrarily; Friedel pairs were merged.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

We are very grateful to Professor Jian-Ming Gu of the Center of Analysis and Measurement of Zhejiang University for help with the X-ray diffraction experiment.

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
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 Comb. Sci.* **24**, 218–226.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC & Rigaku (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yan, J. Z., Xia, C. C. & Rao, G. W. (2006). *Acta Cryst.* E**62**, o1586–o1587.