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13a-Methyl-2,3,3a,3b,11,11a,11b,12,13,13a-decahydro-1*H*-7-azaindeno[5,4-*a*]anthracen-1-ol

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The molecule of the title compound, $C_{21}H_{25}NO$, is built up from five fused rings, four of which are six-membered and one five-membered. O—H···N hydrogen bonds link the molecules into chains and weak C—H···O interactions connect these chains.

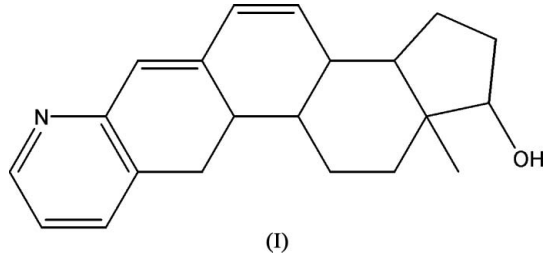
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.037
wR factor = 0.105
Data-to-parameter ratio = 8.5

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

Comment

Testosterone derivatives exhibit a high level 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 have isolated the title product, (I), of the reaction of propargylamine and 17-hydroxyoestra-4,6-dien-3-one as colourless crystals suitable for X-ray analysis.



(I)

The molecular structure of (I) is built up from five fused rings, four of which are six-membered and one five-membered (Fig. 1). The C14–C17/C8/C7 ring fused with the five-membered ring has a chair conformation, as indicated by the puckering parameters θ and φ with values of 5.9 (4)° and −63 (4)°, respectively (Cremer & Pople, 1975). Atoms C1, C2, C3, C4, N1, C9, C10 and C13 are coplanar to within 0.0315 Å, and atoms C11 and C12 deviate from this plane by 0.297 (5) and 0.686 (5) Å, respectively.

The most intersting feature of the structure of (I) is the occurrence of O—H···N hydrogen bonds linking the molecules into chains running along the *b* axis. Weak C—H···O interactions connect these chains (Table 1, Fig. 2).

Experimental

The title compound was prepared according to the procedure of Wang *et al.* (2003). 17-Hydroxyoestra-4,6-dien-3-one (1.36 g, 5 mmol), propargylamine (0.55 g, 10 mmol) and $Cu(NO_3)_2{\cdot}3H_2O$ (0.036 g 0.15 mmol) were added to absolute ethanol (25 ml) with stirring. The mixture was refluxed with stirring at 351 K for 12 h, and then cooled to room temperature and filtered. The filtrate was concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel, eluting with petroleum ether(303–333 K)–diethyl

ether, to give the product as a pale-yellow solid (0.526 g, 31%). A solution of the compound in ethanol was concentrated gradually at room temperature to afford colourless prisms (m.p. 516–528 K).

Crystal data

$C_{21}H_{25}NO$	$Z = 4$
$M_r = 307.42$	$D_x = 1.171$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 11.071$ (3) Å	$\mu = 0.07$ mm^{-1}
$b = 12.118$ (3) Å	$T = 298$ (2) K
$c = 12.998$ (2) Å	Prism, colourless
$V = 1743.8$ (7) Å^3	0.40 × 0.40 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1800 independent reflections
	1008 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.020$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$\theta_{max} = 25.2°$
	3 standard reflections
$T_{min} = 0.972$, $T_{max} = 0.979$	frequency: 60 min
1966 measured reflections	intensity decay: 0.3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.035P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.037$	
$wR(F^2) = 0.105$	
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
1800 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
211 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97* (Sheldrick, 1997)
	Extinction coefficient: 0.019 (2)

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	2.832 (4)	164
C1—H1A···O1^{ii}	0.93	2.43	3.345 (5)	169

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

H atoms were included in calculated positions and treated as riding on their parent atoms, with C—H distances of 0.96 (C_{methyl}), 0.97 ($C_{methylene}$), 0.98 ($C_{methine}$) and 0.93 Å ($C_{aromatic}$) and O—H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}$(parent) for $C_{aromatic}$, $C_{methylene}$, $C_{methine}$ and O, or $1.5U_{eq}$(parent) for C_{methyl}. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The absolute configuration was assigned arbitrarily.

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* (Farrugia, 1997); *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

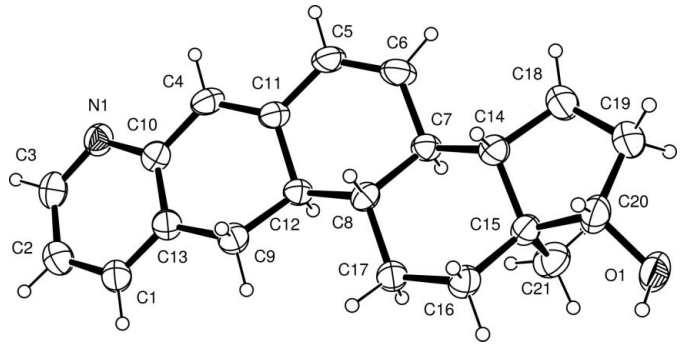


Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

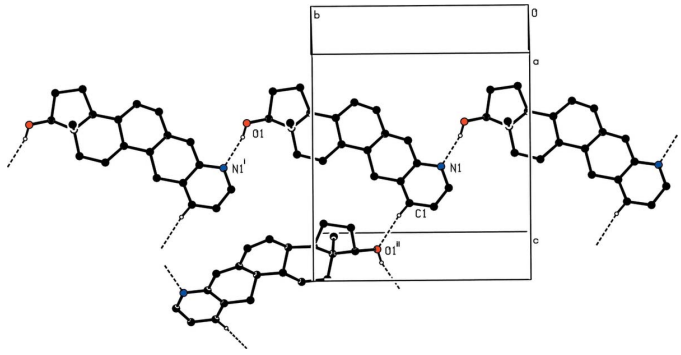


Figure 2
View showing the hydrogen-bonding interactions. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted. [Symmetry codes: (i) $x, 1+y, z$; (ii) $\frac{1}{2}-x, 2-y, \frac{1}{2}+z$.]

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