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17-Isopropyl-3-methoxy-13-methyl-7,8,9,-11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthrene

Huub Kooijman,[a]* Florence C. E. Sarabèr,[b] Aede de Groot[b] and Anthony L. Spek[a]

[a]Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands, and [b]Laboratory of Organic Chemistry, Wageningen University, Dreijenplein 8, 6703 HB Wageningen, The Netherlands

Correspondence e-mail: h.kooijman@chem.uu.nl

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.131
Data-to-parameter ratio = 11.5

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

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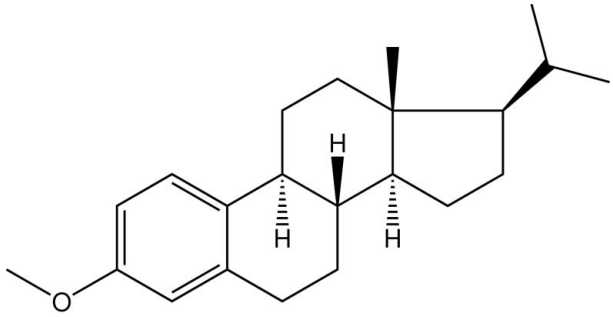
The steroid ring *B* of the title compound, $C_{22}H_{32}O$, is in a half-chair conformation. The five-membered ring is in an intermediate form between the envelope and half-chair conformations.

Comment

The structure of the title compound, (I), was determined in the course of our investigations toward the synthesis of (D-homo) steroid skeletons using Mukaiyama reactions (Sarabèr *et al.*, 2005). The molecular structure of (I) is displayed in Fig. 1. Selected geometric parameters are given in Table 1.



(I)

The six-membered ring containing atom C6 (the steroid ring *B*) is in a half-chair conformation, with the local twofold rotation axis running through the mid-point of the C7—C8 bond, as is illustrated by the asymmetry parameter ΔC_2(C7—C8) = 2.9 (2)° (Duax & Norton, 1975). All other asymmetry parameters have values above 23°. The Cremer and Pople puckering parameters for this ring are θ = 48.7 (2)° and φ = 153.6 (3)° (Cremer & Pople, 1975). Ideal values for this

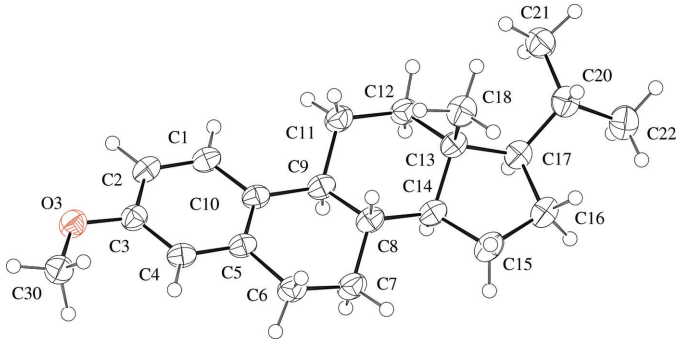


Figure 1
Atomic displacement plot (Spek, 2003) of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

particular half-chair conformation are $\theta = 50.8^\circ$ and $\varphi = 150^\circ$. The six-membered ring containing C11 (the steroid ring *C*) is in a chair conformation, with all asymmetry parameters of type $\Delta C_2(X-Y)$ and $\Delta C_s(X)$ below 8° and all other asymmetry parameters above 111°. The Cremer and Pople θ parameter is 5.46 (19)°; an ideal chair has $\theta = 0^\circ$. The five-membered ring (the steroid ring *D*) is in an intermediate conformation between half-chair [ΔC_2(C13—C14) = 10.83 (18)°] and envelope [ΔC_s(C13) = 9.51 (17)°]. The Cremer and Pople φ parameter is 188.7 (3)°, half-way between the ideal values of 180 (C13-envelope) and 198° (C13—C14 half-chair). The methoxy group is coplanar with the aromatic six-membered ring, as is illustrated by the torsion angle C4—C3—O3—C30 of 4.6 (2)°.

Experimental

The synthesis of the title compound will be described elsewhere (Sarabèr *et al.*, 2005). Crystals suitable for diffraction experiments were obtained by evaporation of a solution of the title compound in methanol.

Crystal data

$C_{22}H_{32}O$
$M_r = 312.48$
Monoclinic, $P2_1/c$
$a = 21.260$ (6) Å
$b = 6.1189$ (10) Å
$c = 13.662$ (3) Å
$\beta = 90.350$ (10)°
$V = 1777.2$ (7) Å^3
$Z = 4$
$D_x = 1.168$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 457 reflections
$\theta = 2.0$–20.0°
$\mu = 0.07$ mm^{-1}
$T = 150$ K
Plate, colourless
0.30 × 0.30 × 0.07 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans and ω scans with κ offset
Absorption correction: none
37859 measured reflections
3508 independent reflections
2308 reflections with $I > 2\sigma(I)$
$R_{int} = 0.097$
$\theta_{max} = 26.0^\circ$
$h = -26 \rightarrow 26$
$k = -7 \rightarrow 7$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.131$
$S = 1.02$
3508 reflections
304 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.13P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—C3	1.376 (2)	O3—C30	1.432 (2)
C3—O3—C30	117.24 (13)		

The coordinates of all H atoms were refined freely. Their displacement parameters were coupled to their carrier atoms according to U_{iso}(H) = 1.5U_{eq}(methyl C) or U_{iso}(H) = 1.2U_{eq}(other C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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