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Key indicators

Single-crystal X-ray study
 T = 150 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 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>.

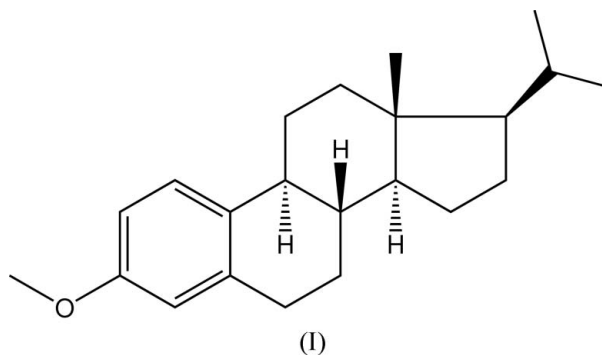
17-Isopropyl-3-methoxy-13-methyl-7,8,9-, 11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthrene

The steroid ring *B* of the title compound, C₂₂H₃₂O, is in a half-chair conformation. The five-membered ring is in an intermediate form between the envelope and half-chair conformations.

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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.



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 $\Delta C_2(\text{C7}-\text{C8}) = 2.9 (2)^\circ$ (Duax & Norton, 1975). All other asymmetry parameters have values above 23° . The Cremer and Pople puckering parameters for this ring are $\theta = 48.7 (2)^\circ$ and $\varphi = 153.6 (3)^\circ$ (Cremer & Pople, 1975). Ideal values for this

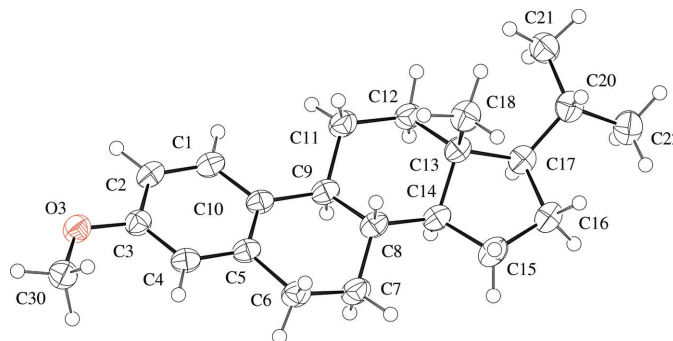


Figure 1 Atomic displacement plot (Spek, 2003) of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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 $5.46(19)^\circ$; an ideal chair has $\theta = 0^\circ$. The five-membered ring (the steroid ring D) is in an intermediate conformation between half-chair [$\Delta C_2(C13-C14) = 10.83(18)^\circ$] and envelope [$\Delta C_s(C13) = 9.51(17)^\circ$]. The Cremer and Pople φ parameter is $188.7(3)^\circ$, 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 $4.6(2)^\circ$.

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

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

Data collection

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

Refinement

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

Table 1

Selected geometric parameters (\AA , $^\circ$).

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 $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ or $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{other C})$.

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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