Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,6-Dimethyl-3-methylenebicyclo[3.2.1]octane-2,8-diol

Simon J. Coles* and Michael B. Hursthouse

Department of Chemistry, University of Southampton, Southampton SO17 1BJ, England

Correspondence e-mail: s.j.coles@soton.ac.uk

Received 3 May 2002
Accepted 13 May 2002
Online 24 May 2002

The title compound, $C_{11}H_{18}O_2$, is a natural product mimic containing five chiral centres.

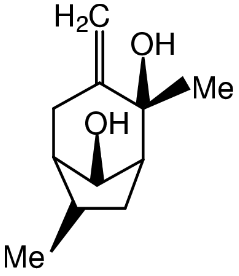
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.123
Data-to-parameter ratio = 18.4

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

Comment

The title compound, (I), was synthesized as part of a research programme investigating synthetic routes to bicyclo[3.2.1]-octanes, which are a common structural component of many natural products, such as kaurenoids and gibberellins.



(I)

The bond lengths and angles in (I) conform to standard values (Allen *et al.*, 1987) derived from the Cambridge Structural Database (Allen & Kennard, 1993). The bicyclic structure of (I) produces three ring systems, in which the five-membered ring is in an envelope conformation, with C7 as the flap, the six-membered ring is in a chair conformation, and the seven-membered ring is in a boat conformation. The hydroxyl substituents on the six-membered ring are both in axial positions on the same side of the molecule, while the methyl and methene substituents are in equatorial positions. The methyl group located on the five-membered ring is in an axial position. The molecule has five chiral centres, with C2, C6 and C7 of one chirality, and C8 and C10 the opposite.

There are two classical hydrogen bonds in the crystal structure. An intramolecular interaction with O···O = 2.5840 (17) Å exists for O1—H1···O2, and there is an O···O separation of 2.7000 (15) Å in the intermolecular interaction O2—H2···O1^{i} [symmetry code: (i) $x-1/2$, $1/2-y$, $z-1/2$]. This intermolecular interaction gives rise to a one-dimensional chain motif in the crystal structure.

Experimental

The title compound was prepared by cyclization of the appropriate methylenecyclopropyl ketone to form the bicyclo[3.2.1]octane. The cyclization was mediated by slow addition of the ketone to two equivalents of SmI_2 in the presence of ^{t}BuOH (2 equivalents) and HMPA (10 equivalents) in THF at 273 K.

DOI: 10.1107/S1600536802008644

Crystal data

$C_{11}H_{18}O_2$
$M_r = 182.25$
Monoclinic, $P2_1/n$
$a = 6.8216\ (14)$ Å
$b = 18.934\ (4)$ Å
$c = 8.1610\ (16)$ Å
$\beta = 107.76\ (3)°$
$V = 1003.9\ (3)$ Å^3
$Z = 4$

$D_x = 1.206$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5108 reflections
$\theta = 2.9$–$30.5°$
$\mu = 0.08$ mm^{-1}
$T = 150\ (2)$ K
Needle, colourless
0.40 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.968$, $T_{\max} = 0.996$
7926 measured reflections

2262 independent reflections
1447 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 8$
$k = -23 \rightarrow 24$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.123$
$S = 0.99$
2262 reflections
123 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2]$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.011 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.84	1.83	2.5840 (17)	149
O2—H2···O1$^{\rm i}$	0.84	1.87	2.7000 (15)	170

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

H atoms were refined with constrained positions and with $U_{\rm iso}$ tied to $U_{\rm eq}$ of their parent atoms. The hydroxyl H atoms were allowed torsional freedom.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

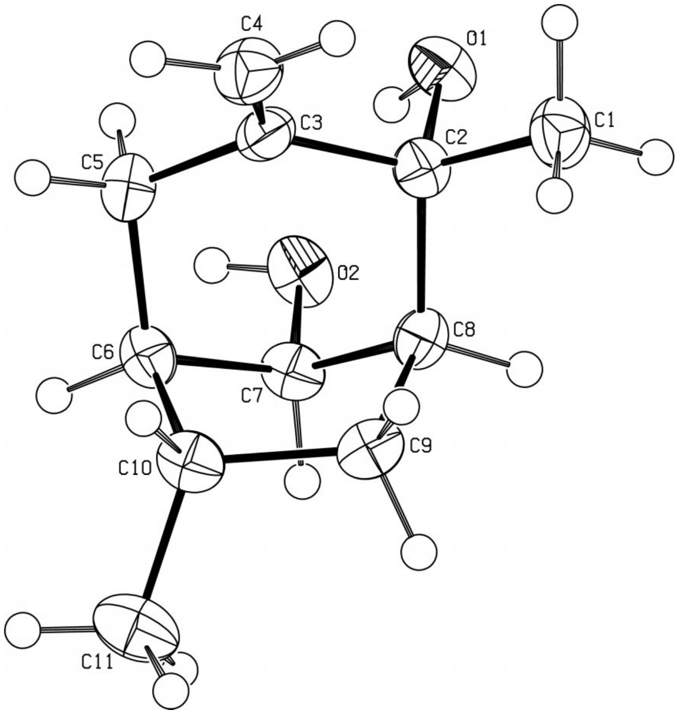


Figure 1
View of (I) (50% probability displacement ellipsoids).

The authors thank the EPSRC for funding crystallographic facilities.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.