

Table 2. Selected geometric parameters (Å, °)

O1—C2	1.386 (3)	C6—C11	1.410 (3)
O1—C13	1.368 (2)	O7—C8	1.388 (2)
C5—C10	1.418 (3)	O7—C11	1.375 (2)
O1—C2—C3	116.0 (2)	C4—C10—C5	124.4 (2)
O1—C2—O2	116.7 (2)	C6—C11—C12	126.8 (2)
O2—C2—C3	127.2 (3)	C11—C12—C13	117.8 (2)
C6—C5—C10	124.4 (2)	O1—C13—C12	118.0 (2)
C5—C6—C11	112.0 (2)		
<i>D</i> —H... <i>A</i>	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C16—H163...O7	2.31 (3)	2.943 (3)	122 (2)
C9—H9...O22 ⁱ	2.60 (2)	3.155 (3)	118 (2)
C21—H211...O2 ⁱⁱ	2.79 (5)	3.286 (4)	114 (3)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, 1+y, z$.

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. Isotropic refinement of the structure by least-squares methods was followed by anisotropic refinement of all the non-H atoms. All H atoms were located from a difference Fourier map and their positions and isotropic displacement parameters were refined (except U_{iso} of H213). All calculations were performed on a PC/AT computer.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local program. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: DE1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-3-ol

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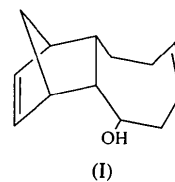
(Received 11 April 1995; accepted 14 July 1995)

Abstract

The cyclooctenyl fragment of the title compound, $C_{13}H_{18}O$, adopts a twist-boat conformation. The torsion angle at the ring-fusion bond is $7.0(2)^\circ$ and that at the cyclooctene double bond is $0.0(2)^\circ$. The cyclooctenyl C=C bond length is $1.315(3)$ Å and the norbornenyl C=C bond length is $1.323(2)$ Å. In accord with the space-group symmetry, four molecules are held together by hydrogen bonding, with an intermolecular O...O distance of $2.736(1)$ Å.

Comment

The bond lengths and angles C7—C12, C9—C10, C1—C12—C11 and C6—C7—C8 (listed in Table 3) of tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-3-ol, (I), are similar to those of tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-one, (II) (Garcia, Fronczek & McLaughlin 1991), *endo*-13-*syn*-methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-ol, (III) (Garcia, Morales, Fronczek & McLaughlin, 1994), and *endo*-13-*syn*-methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-yl acetate, (IV) (Garcia, Morales, Fronczek & McLaughlin 1994).



There is no evidence that the hydroxy group has any influence on the conformation of the cyclooctenyl ring. The atom positions of the comparable known crystal structures were taken from the Cambridge Structural Database (1995) and transferred into *SYBIL6.1* (Tripos

Associates Inc., 1994). The matching of the C₁₃ skeleton of the title compound with those of tricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-one and *endo*-13-*syn*-methyltricyclo[8.2.1.0^{2,9}]trideca-5,11-dien-13-*anti*-yl acetate shows only small r.m.s. deviations.

The cyclooctene ring adopts a twist-boat conformation with torsion angles of 7.0(2)° at the ring fusion bond and 0.0(2)° at the C3=C4 double bond of the cyclooctene ring. Corresponding torsion angles of the known compounds (II), (III) and (IV) are -2.8(2) and 2.5(2)° (Garcia, Fronczek & McLaughlin 1991),

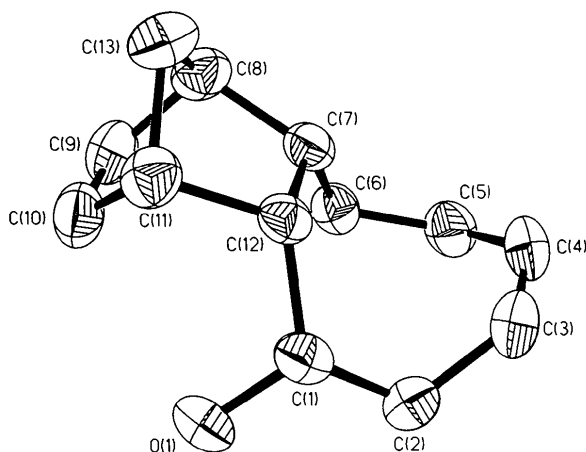


Fig. 1. The molecular structure (SHELXTL-Plus; Sheldrick, 1989) of the title compound, showing 50% probability ellipsoids (H atoms have been omitted for clarity).

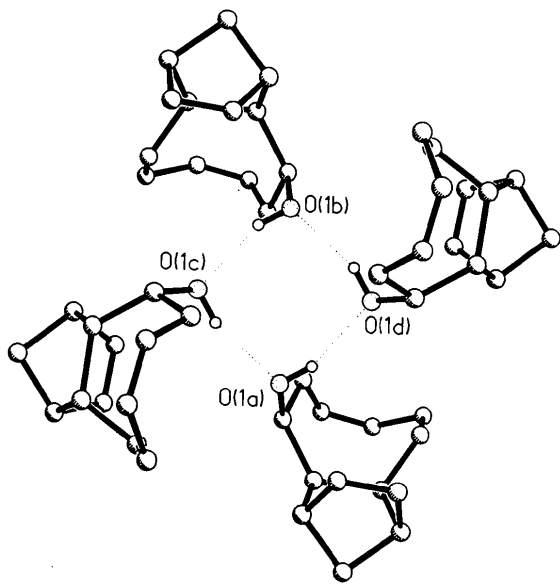


Fig. 2. Aggregation of four molecules by hydrogen bonding. Hydrogen bonds are illustrated by dotted lines.

-1.1(4) and 1.5(6)° (Garcia, Morales, Fronczek & McLaughlin 1994), and -0.1(3) and -1.4(4)° (Garcia, Morales, Fronczek & McLaughlin 1994), respectively. In accord with the space-group symmetry, four molecules are held together by hydrogen bonding (Fig. 2). The values of the O...O distance and the O...O...O angle are 2.736(1) Å and 89.2(1)°, respectively.

Experimental

The title compound, (I), was synthesized by borontrifluorate-etherate-catalyzed Diels-Alder reaction (Oppholzer, 1984) of 2,5-cyclooctadienone (Cantrell & Solomon, 1970) and cyclopentadiene followed by reaction of the keto group with sodium borontrifluoride (Hudlicky, 1984). The crystals were grown in diethyl ether (m.p. 364.8 K).

Crystal data

C₁₃H₁₈O
M_r = 190.3
 Tetragonal
*I*4
a = 15.515(1) Å
c = 8.851(1) Å
V = 2130.7(2) Å³
Z = 8
D_x = 1.19 Mg m⁻³

Mo Kα radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 13.16–19.19°
 μ = 0.068 mm⁻¹
T = 298 K
 Rectangular
 0.63 × 0.46 × 0.46 mm
 Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 5047 measured reflections
 2438 independent reflections
 2241 observed reflections
 $[I > 2\sigma(I)]$

R_{int} = 0.01
 θ_{\max} = 27.45°
h = 0 → 21
k = 0 → 21
l = 0 → 12
 3 standard reflections monitored every 100 reflections
 intensity decay: <2%

Refinement

Refinement on *F*²
R(*F*) = 0.034
 $wR(F^2)$ = 0.102
S = 0.82
 2438 reflections
 128 parameters
 H atoms refined using a riding model

$w = 1/[\sigma^2(F_o^2) + (0.100P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.06$
 $\Delta\rho_{\max} = 0.156 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.183 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
C1	0.3288 (1)	0.0493 (1)	0.1031 (1)	0.038 (1)
C2	0.3721 (1)	0.0053 (1)	-0.0330 (1)	0.049 (1)
C3	0.3140 (1)	-0.0094 (1)	-0.1657 (2)	0.058 (1)

C4	0.2707 (1)	-0.0812 (1)	-0.1874 (1)	0.060 (1)
C5	0.2714 (1)	-0.1584 (1)	-0.0851 (2)	0.053 (1)
C6	0.2541 (1)	-0.1414 (1)	0.0840 (1)	0.042 (1)
C7	0.1934 (1)	-0.0655 (1)	0.1140 (1)	0.039 (1)
C8	0.1405 (1)	-0.0746 (1)	0.2637 (2)	0.051 (1)
C9	0.2041 (1)	-0.0820 (1)	0.3912 (2)	0.053 (1)
C10	0.2409 (1)	-0.0058 (1)	0.4079 (1)	0.052 (1)
C11	0.2016 (1)	0.0545 (1)	0.2922 (2)	0.046 (1)
C12	0.2333 (1)	0.0272 (1)	0.1313 (1)	0.035 (1)
C13	0.1096 (1)	0.0188 (1)	0.2872 (2)	0.058 (1)
O1	0.3826 (1)	0.0394 (1)	0.2321 (1)	0.044 (1)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.539 (2)	C1—C12	1.542 (1)
C1—O1	1.423 (1)	C2—C3	1.499 (2)
C3—C4	1.315 (3)	C4—C5	1.503 (2)
C5—C6	1.544 (2)	C6—C7	1.531 (2)
C7—C8	1.565 (2)	C7—C12	1.574 (2)
C8—C9	1.504 (2)	C8—C13	1.540 (2)
C9—C10	1.323 (2)	C10—C11	1.515 (2)
C11—C12	1.566 (2)	C11—C13	1.533 (2)
O1—O1	2.736 (1)		
O1—C1—C12	114.3 (1)	O1—C1—C2	108.9 (1)
C12—C1—C2	116.6 (1)	C3—C2—C1	114.7 (1)
C4—C3—C2	123.5 (1)	C5—C4—C3	125.7 (1)
C6—C5—C4	116.5 (1)	C7—C6—C5	114.0 (1)
C12—C7—C8	101.9 (1)	C12—C7—C6	118.5 (1)
C8—C7—C6	113.6 (1)	C13—C8—C9	100.1 (1)
C13—C8—C7	101.2 (1)	C9—C8—C7	107.4 (1)
C10—C9—C8	107.4 (1)	C11—C10—C9	107.6 (1)
C13—C11—C10	99.7 (1)	C13—C11—C12	99.9 (1)
C12—C11—C10	108.8 (1)	C11—C12—C7	102.3 (1)
C11—C12—C1	112.9 (1)	C7—C12—C1	124.5 (1)
C11—C13—C8	93.1 (1)		

Table 3. Comparison of selected bond lengths (Å) and bond angles (°) of known crystal structures

	C7—C12	C9—C10	C1—C12—C11	C6—C7—C8
(I)	1.574 (2)	1.323 (2)	112.9 (1)	113.6 (1)
(II)	1.571 (2)	1.318 (2)	110.8 (1)	113.6 (1)
(III) (molecule 1)	1.564 (3)	1.307 (4)	112.4 (2)	112.9 (2)
(molecule 2)	1.561 (4)	1.313 (5)	113.1 (2)	112.2 (3)
(IV)	1.569 (2)	1.312 (2)	112.3 (2)	111.8 (1)

Table 4. Comparison of r.m.s. deviations (Å) of the C₁₃ skeleton of tricyclo[8.2.1.0^{2,9}]trideca-5,11-diene in known crystal structures

	(I)	(II)	(III)	(IV)
R.m.s. deviation	0.09	0.17	0.09	0.21

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovitch, 1965). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993), *GFMLX* (Flack, 1983). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1989), *XANADU* (Roberts & Sheldrick, 1975), *SYBIL* (Tripos Associates Inc., 1994). Software used to prepare material for publication: *DAESD* (Davis & Harris, 1970).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1181). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Benzoyl-2-phenyldecahydroquinolin-4-one, C₂₂H₂₃NO₂

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Abstract

The title molecule consists of a piperidin-4-one ring fused to a cyclohexane moiety. The piperidine ring adopts a twisted conformation and the cyclohexane ring adopts a chair conformation. The crystal structure determination confirms the *trans* fusion of the two rings.

† Deceased.