C21	0.5826 (15)	0.2291 (3)	0.3948 (5)	0.098 (6)
C22	0.7849 (14)	0.2608 (3)	0.4079 (6)	0.097 (6)
C23	0.9193 (13)	0.2591 (4)	0.4922 (7)	0.106 (6)
C24	0.8519 (12)	0.2258 (3)	0.5645 (5)	0.084 (5)

Table 2. *Geometric parameters* (Å, °)

S—O17	1.442 (5)	C8—C13	1.509 (8)
S—O18	1.422 (5)	C8—C14	1.541 (8)
S—C14	1.795 (6)	C10—C11	1.501 (11)
S—C19	1.766 (7)	C11—C12	1.519 (11)
O1—C2	1.418 (9)	C12—C13	1.518 (9)
O1—C6	1.407 (7)	C14—C15	1.534 (12)
O7—C6	1.442 (7)	C14—C15*A*	1.587 (17)
O7—C8	1.425 (7)	C15—C16	1.530 (13)
O9—C8	1.406 (6)	C15*A*—C16*A*	1.507 (24)
O9—C10	1.424 (8)	C19—C20	1.354 (10)
C2—C3	1.487 (11)	C19—C24	1.368 (9)
C3—C4	1.527 (13)	C20—C21	1.385 (10)
C4—C5	1.480 (17)	C21—C22	1.361 (12)
C5—C6	1.480 (10)	C22—C23	1.357 (13)
C6—C16	1.623 (11)	C23—C24	1.376 (11)
C6—C16*A*	1.416 (21)		
O17—S—O18	118.9 (4)	O9—C8—C13	112.9 (5)
O17—S—C14	110.7 (3)	O9—C8—C14	105.6 (4)
O17—S—C19	106.8 (3)	C13—C8—C14	110.4 (5)
O18—S—C14	109.5 (3)	O9—C10—C11	111.7 (5)
O18—S—C19	107.6 (3)	C10—C11—C12	110.3 (5)
C14—S—C19	101.9 (3)	C11—C12—C13	109.6 (6)
C2—O1—C6	115.8 (5)	C8—C13—C12	111.9 (5)
C6—O7—C8	119.9 (4)	S—C14—C8	114.2 (4)
C8—O9—C10	115.1 (4)	S—C14—C15	112.8 (5)
O1—C2—C3	112.3 (7)	S—C14—C15*A*	101.8 (6)
C2—C3—C4	106.5 (6)	C8—C14—C15	117.4 (6)
C3—C4—C5	110.6 (7)	C8—C14—C15*A*	104.4 (7)
C4—C5—C6	113.8 (8)	C14—C15—C16	105.8 (7)
O1—C6—O7	110.2 (5)	C14—C15*A*—C16*A*	109.3 (12)
O1—C6—C5	109.8 (5)	C6—C16—C15	107.4 (7)
O1—C6—C16	97.4 (5)	C6—C16*A*—C15*A*	109.6 (14)
O1—C6—C16*A*	127.2 (9)	S—C19—C20	121.3 (5)
O7—C6—C5	106.3 (6)	S—C19—C24	118.6 (5)
O7—C6—C16	110.6 (5)	C20—C19—C24	120.0 (6)
O7—C6—C16*A*	109.7 (9)	C19—C20—C21	119.8 (7)
C5—C6—C16	122.2 (7)	C20—C21—C22	120.2 (7)
C5—C6—C16*A*	90.5 (9)	C21—C22—C23	119.6 (7)
O7—C8—O9	112.4 (4)	C22—C23—C24	120.5 (7)
O7—C8—C13	105.5 (4)	C19—C24—C23	119.7 (7)
O7—C8—C14	110.2 (5)		

The θ-scan width was $(0.80 + 0.33\tan\theta)^\circ$. Background measurements were extended by 25% of the peak scan on either side of each peak. The maximum time for measurement of one reflection was 60 s. Data collection and cell refinement: Enraf-Nonius (1984) *CAD*-4 *PDP*11 *Software*. Data reduction: *NRCVAX DATRD*2 (Gabe, Le Page, Charland, Lee & White, 1989). Program used to solve structure: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Software used to prepare material for publication: *NRCVAX TABLES*; *ORTEP*II (Johnson, 1976). Program used to refine structure: *NRCVAX LSTSQ*. Block-diagonal least-squares refinement was performed, with H atoms included in their calculated positions, but not refined. Isotropic U values were assigned to the H atoms as equal to $U_{\rm eq}$ of the attached atom plus 0.010 Å^2. C15 and C16 of the central ring, along with their attached H atoms, were found to be disordered, with site occupancy factors of 0.65 and 0.35 for the major and minor conformers, respectively. Relative amounts of the two forms were determined by refining the population parameters of C15, C16, C15*A* and C16*A*, while holding the temperature factors constant. C15, C16, C15*A* and C16*A* were given isotropic thermal parameters during refinement; all other non-H atoms were given anisotropic thermal parameters. In the final refinement, the population parameters for C15, C16, H151, H152, H161 and H162 were set at 0.65 and the population parameters for C15*A*, C16*A*, H153, H154, H163 and H164 were set at 0.35.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71275 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1033]

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Structures of Two Hexacyclic Cage Diketone Monoacetals

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Abstract

The structures of 10,10-(ethylenedioxy)hexacyclo[10.2.1.0^{2,11}.0^{4,9}.0^{4,14}.0^{9,13}]pentadeca-5,7-dien-3-one and 10,10-diisopropoxyhexacyclo[10.2.1.0^{2,11}.0^{4,9}.0^{4,14}.0^{9,13}]pentadeca-5,7-dien-3-one are reported. In each structure the cyclohexadiene ring is planar. Bonding geometries are similar to those found in other cage structures, except for elongations of the

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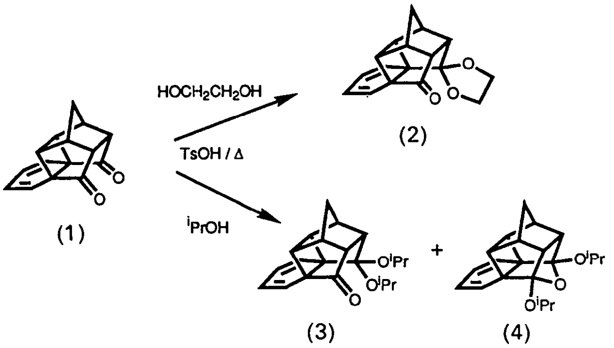
C4—C9 propellane-type bonds due to the fusion of the diene rings. A slight flattening of the boat conformations of the unbridged six-membered rings is observed in the acetals relative to the parent dione.

Comment

As part of a program that involves the synthesis and chemistry of novel substituted cage compounds (Marchand, 1989, 1991), we undertook to synthesize a series of unsymmetrically substituted hexacyclo-$[10.2.1.0^{2,11}.0^{4,9}.0^{4,14}.0^{9,13}]$pentadeca-5,7-dienes in order to determine the effects of remote substituents on the π-facial selectivities of Diels–Alder reactions (Coxon, O'Connell & Steel, 1987; Coxon, Maclagan, McDonald & Steel, 1991). The readily available 3,10-dione (1) (Kushner, 1971; Pandey, Zope & Ayyangar, 1990) is a useful starting material for this purpose. In order to defeat the twofold symmetry inherent in (1), we prepared monoacetals of (1) by reactions with alcohols. Thus, reaction with ethanediol cleanly affords the monoethyleneacetal (2) while reaction with isopropanol produces a mixture of the unsymmetrical monoacetal (3) along with a small amount of the transannularly cyclized bisacetal (4), as shown in the scheme below. In order to confirm the structures and to examine the steric environments of the diene moieties in these compounds, X-ray structure determinations were carried out.



The structures of (2) and (3) are confirmed by the X-ray analyses as shown in Figs. 1 and 2. Each compound consists of the pentacycloundecane cage system (Marchand, 1989) in which the open side of the cage is functionalized by ketone and acetal groups and the diene ring is fused across the adjacent cyclobutane ring junction. In both structures the cyclohexadiene ring is planar to within 0.02 Å. As has been noted for previously reported cage structures (Flippen-Anderson, Gilardi, George, Marchand & Reddy, 1989, and references therein), C—C bonds involving C3, C10 or C15 are significantly shortened and the C2—C11 bonds are lengthened relative to the other (normal) bond lengths. The only exception to the usual pattern is the slight lengthening of the C4—C9 propellane-type bonds due to the presence of the diene ring. This lengthening is also observed in the structures of the parent dione (1) (Dhaneshwar, Tavale, Guru Row, Zope, Pandey & Ayyangar, 1988) and three of its Diels–Alder adducts (Coxon, O'Connell & Steel, 1986).

The greater steric demand of the acetal groups results in a slight lengthening of the C3—C10 nonbonded distance in (2) [2.683 (6) Å] and in (3) [2.696 (8) Å] relative to the corresponding distance in the parent dione (1) [2.563 (5) Å]. This corresponds to a flattening of the boat conformation of the six-membered ring and is also observed in the structures of other cage diketone monoacetals (Flippen-Anderson, Gilardi, George, Marchand & Reddy, 1989; Watson, Nagl, Marchand, Reddy & Reddy, 1989).

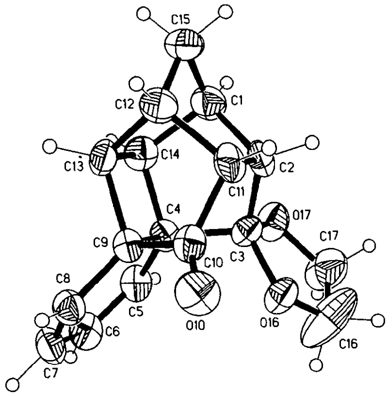


Fig. 1. Perspective view and atom labelling of (2). Thermal ellipsoids are shown at 50% probability levels; H atoms as circles of arbitrary radii.

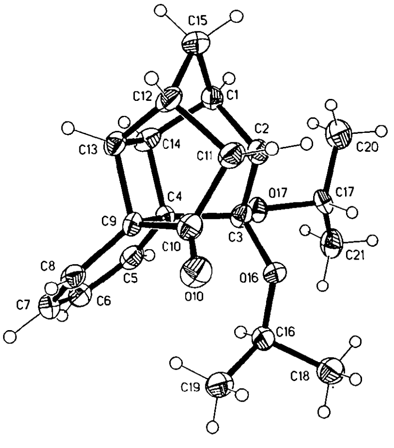


Fig. 2. Perspective view and atom labelling of (3). Thermal ellipsoids are shown at 50% probability levels; H atoms as circles of arbitrary radii.

Experimental

Compound (2)

Crystal data

$C_{17}H_{16}O_3$
M_r = 268.3
Orthorhombic
Pbcn
a = 21.972 (2) Å
b = 9.2992 (6) Å
c = 12.486 (1) Å
V = 2551.2 (7) Å^3
Z = 8
D_x = 1.397 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 18–20°
μ = 0.089 mm^{-1}
T = 294 K
Block
0.5 × 0.3 × 0.2 mm
Colourless
Crystal source: recrystallized from ethyl acetate–hexane

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical (Walker & Stuart, 1983)
$T_{\min}$ = 0.88, $T_{\max}$ = 1.22
1857 measured reflections
1857 independent reflections
959 observed reflections $[I \geq 3\sigma(I)]$
$\theta_{\max}$ = 22°
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity variation: $\leq$1.4%

Refinement

Refinement on F
Final R = 0.044
wR = 0.045
S = 1.07
959 reflections
181 parameters
H-atom parameters not refined
$w = [\sigma^2(F_o) + 0.04(F_o)^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.005
$\Delta\rho_{\max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\min}$ = −0.33 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (3)

Crystal data

$C_{21}H_{26}O_3$
M_r = 326.4
Triclinic
$P\overline{1}$
a = 8.726 (6) Å
b = 10.013 (5) Å
c = 10.322 (7) Å
α = 96.26 (5)°
β = 109.63 (5)°
γ = 91.24 (5)°
V = 842.8 (9) Å^3
Z = 2
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 15 reflections
θ = 7.5–15°
μ = 0.079 mm^{-1}
T = 175 K
Block
0.36 × 0.16 × 0.06 mm
Colourless
Crystal source: recrystallized from ether–petroleum ether

Data collection

Nicolet $R3m$ diffractometer
ω scans
Absorption correction: none
3147 measured reflections
R_{int} = 0.030
$\theta_{\max}$ = 25°
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 12$
2968 independent reflections
1098 observed reflections $[I \geq 3\sigma(I)]$
3 standard reflections monitored every 100 reflections
intensity variation: $\leq$2.5%

Refinement

Refinement on F
Final R = 0.051
wR = 0.052
S = 1.18
1098 reflections
217 parameters
H-atom parameters not refined
$w = [\sigma^2(F_o) + 0.0005(F_o)^2]^{-1}$
$(\Delta/\sigma)_{\max}$ = 0.002
$\Delta\rho_{\max}$ = 0.21 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e 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 thermal parameters* (Å^2) *for* (2)

$B_{\text{eq}} = (4/3)\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O10	0.1970 (2)	0.2126 (4)	0.1283 (3)	3.9 (1)
O16	0.1611 (1)	0.1228 (4)	−0.1123 (2)	3.3 (1)
O17	0.1179 (2)	0.2472 (4)	−0.2502 (3)	3.6 (1)
C1	0.1425 (2)	0.5163 (6)	−0.1450 (3)	3.4 (1)
C2	0.1822 (2)	0.3815 (5)	−0.1281 (4)	2.8 (1)
C3	0.1371 (2)	0.2579 (5)	−0.1410 (3)	2.6 (1)
C4	0.0843 (2)	0.3060 (5)	−0.0702 (3)	2.4 (1)
C5	0.0257 (2)	0.2298 (6)	−0.0860 (4)	3.1 (1)
C6	−0.0065 (2)	0.1766 (5)	−0.0061 (4)	3.3 (1)
C7	0.0142 (2)	0.1861 (6)	0.1042 (4)	3.3 (1)
C8	0.0655 (2)	0.2509 (5)	0.1308 (4)	2.9 (1)
C9	0.1054 (2)	0.3197 (5)	0.0500 (3)	2.3 (1)
C10	0.1736 (2)	0.2900 (5)	0.0627 (4)	2.6 (1)
C11	0.2042 (2)	0.3977 (5)	−0.0089 (4)	2.9 (1)
C12	0.1730 (2)	0.5382 (5)	0.0260 (4)	3.3 (1)
C13	0.1053 (2)	0.4875 (5)	0.0365 (4)	2.8 (1)
C14	0.0848 (2)	0.4718 (5)	−0.0811 (4)	3.0 (1)
C15	0.1713 (2)	0.6307 (6)	−0.0745 (5)	4.1 (1)
C16	0.1615 (3)	0.0372 (7)	−0.2012 (5)	7.4 (2)
C17	0.1386 (3)	0.1154 (7)	−0.2903 (5)	5.7 (2)

Table 2. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (3)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O10	0.5649 (4)	0.5805 (4)	0.8437 (4)	0.031 (2)
O16	0.4796 (4)	0.8838 (3)	0.7936 (4)	0.022 (2)
O17	0.2289 (4)	0.9830 (3)	0.7172 (4)	0.023 (2)
C1	0.0922 (6)	0.7832 (6)	0.8328 (6)	0.021 (2)
C2	0.2770 (6)	0.8198 (5)	0.8855 (6)	0.025 (2)
C3	0.3079 (6)	0.8660 (5)	0.7605 (6)	0.021 (2)
C4	0.2194 (7)	0.7502 (5)	0.6461 (6)	0.024 (2)
C5	0.1800 (6)	0.7726 (6)	0.4988 (7)	0.027 (3)
C6	0.2002 (6)	0.6834 (6)	0.4007 (6)	0.026 (3)
C7	0.2747 (6)	0.5571 (5)	0.4363 (7)	0.024 (2)
C8	0.3228 (6)	0.5238 (5)	0.5629 (6)	0.023 (3)
C9	0.2940 (6)	0.6105 (5)	0.6789 (6)	0.017 (2)
C10	0.4305 (7)	0.6255 (5)	0.8176 (6)	0.023 (2)
C11	0.3537 (7)	0.6819 (5)	0.9230 (6)	0.026 (2)
C12	0.2005 (6)	0.5829 (5)	0.8831 (6)	0.026 (3)
C13	0.1456 (7)	0.5706 (5)	0.7221 (6)	0.024 (2)
C14	0.0706 (6)	0.7065 (5)	0.6885 (6)	0.025 (3)
C15	0.0735 (7)	0.6707 (6)	0.9168 (7)	0.030 (3)
C16	0.5407 (7)	0.9197 (5)	0.6871 (6)	0.025 (2)
C17	0.2787 (7)	1.1061 (5)	0.8133 (6)	0.021 (2)
C18	0.6668 (7)	1.0380 (5)	0.7510 (6)	0.032 (3)
C19	0.6170 (7)	0.8018 (6)	0.6348 (7)	0.034 (3)
C20	0.1574 (7)	1.1389 (6)	0.8847 (7)	0.037 (3)
C21	0.2907 (7)	1.2161 (6)	0.7262 (6)	0.033 (3)

Table 3. *Bond distances* (Å) *and angles* (°)

	(2)	(3)		(2)	(3)
O10—C10	1.205 (6)	1.220 (7)	C6—C7	1.452 (7)	1.460 (8)
O16—C3	1.409 (6)	1.422 (6)	C7—C8	1.321 (7)	1.315 (9)
O16—C16	1.366 (7)	1.447 (8)	C8—C9	1.481 (6)	1.495 (9)
O17—C3	1.431 (5)	1.411 (7)	C9—C10	1.532 (6)	1.513 (7)
O17—C17	1.401 (7)	1.453 (6)	C9—C13	1.569 (6)	1.562 (9)
C1—C2	1.542 (7)	1.539 (7)	C10—C11	1.502 (6)	1.525 (10)
C1—C14	1.555 (7)	1.549 (9)	C11—C12	1.539 (7)	1.556 (8)
C1—C15	1.520 (7)	1.533 (9)	C12—C13	1.565 (6)	1.556 (9)
C2—C3	1.526 (7)	1.521 (10)	C12—C15	1.521 (7)	1.535 (9)
C2—C11	1.572 (6)	1.577 (8)	C13—C14	1.543 (6)	1.546 (8)
C3—C4	1.526 (6)	1.544 (7)	C16—C17	1.421 (9)	
C4—C5	1.483 (6)	1.485 (9)	C16—C18		1.528 (7)
C4—C9	1.576 (6)	1.579 (8)	C16—C19		1.506 (9)
C4—C14	1.547 (7)	1.571 (9)	C17—C20		1.502 (10)
C5—C6	1.320 (7)	1.336 (9)	C17—C21		1.519 (9)
C3—O16—C16	108.4 (4)	118.2 (4)	C8—C9—C10	115.5 (4)	116.5 (5)
C3—O17—C17	107.8 (4)	117.1 (4)	C8—C9—C13	120.1 (4)	118.2 (4)
C2—C1—C14	100.1 (4)	100.2 (5)	C10—C9—C13	101.1 (3)	101.0 (5)
C2—C1—C15	104.7 (4)	104.0 (4)	O10—C10—C9	126.6 (4)	127.2 (6)
C14—C1—C15	103.2 (4)	103.1 (4)	O10—C10—C11	127.7 (4)	126.2 (5)
C1—C2—C3	103.3 (4)	103.8 (4)	C9—C10—C11	104.8 (4)	105.4 (5)
C1—C2—C11	103.0 (4)	103.4 (4)	C2—C11—C10	111.2 (4)	110.7 (5)
C3—C2—C11	111.8 (4)	112.5 (5)	C2—C11—C12	102.3 (4)	102.6 (4)
O16—C3—O17	106.9 (3)	111.8 (4)	C10—C11—C12	101.4 (4)	100.4 (4)
O16—C3—C2	113.7 (4)	107.7 (4)	C11—C12—C13	101.1 (4)	100.4 (5)
O16—C3—C4	113.5 (4)	116.4 (5)	C11—C12—C15	104.9 (4)	103.3 (4)
O17—C3—C2	110.1 (4)	113.7 (5)	C13—C12—C15	102.6 (4)	103.3 (4)
O17—C3—C4	110.3 (4)	105.7 (4)	C9—C13—C12	107.9 (4)	109.1 (4)
C2—C3—C4	102.3 (4)	101.3 (5)	C9—C13—C14	90.5 (3)	91.1 (5)
C3—C4—C5	116.3 (4)	118.7 (5)	C12—C13—C14	103.1 (4)	103.3 (5)
C3—C4—C9	110.6 (3)	112.2 (4)	C1—C14—C4	108.4 (4)	108.0 (4)
C3—C4—C14	103.7 (4)	103.1 (5)	C1—C14—C13	103.0 (4)	103.7 (5)
C5—C4—C9	114.9 (4)	113.5 (5)	C4—C14—C13	90.8 (3)	90.1 (4)
C5—C4—C14	118.1 (4)	115.6 (4)	C1—C15—C12	95.3 (4)	95.6 (5)
C9—C4—C14	90.0 (3)	89.6 (4)	O16—C16—C17	109.6 (5)	
C4—C5—C6	123.0 (4)	124.4 (5)	O16—C16—C18		107.7 (5)
C5—C6—C7	121.7 (4)	120.3 (6)	O16—C16—C19		110.2 (5)
C6—C7—C8	122.2 (4)	123.0 (6)	C18—C16—C19		110.5 (5)
C7—C8—C9	122.1 (4)	121.4 (5)	O17—C17—C16	106.4 (5)	
C4—C9—C8	116.1 (4)	117.1 (5)	O17—C17—C20		111.8 (4)
C4—C9—C10	111.8 (3)	110.8 (4)	O17—C17—C21		105.6 (5)
C4—C9—C13	88.7 (3)	89.2 (4)	C20—C17—C21		110.2 (5)

For (2): structure solution by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refinement by full-matrix least squares (Enraf-Nonius, 1990). For (3): structure solution by direct methods and full-matrix least-squares refinement with *SHELXTL-Plus* (Sheldrick, 1991). H atoms were located from difference maps but included in calculated positions. No attempt was made to model the disorder of C16 in (2) suggested by its bonding and thermal parameters.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71265 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1054]

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8-Pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undec-3′-yl-*exo*-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane

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Abstract

The title compound is composed of two $C_{11}H_{14}$ geometric isomers, pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (PCU) and pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (trishomocubane), connected by a single bond. The PCU isomer contains four five-membered rings, a four-membered ring and a six-membered ring fused into a cage-like structure, while the trishomocubane isomer is a fusion of six five-membered rings. Elongated bond lengths in these molecules are compared with literature structures and with the results of molecular-mechanics calculations.

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