C(16′)	−1.3669 (9)	0.5804 (2)	−1.0315 (8)	0.073 (2)
C(17′)	−1.0815 (6)	0.6170 (2)	−0.5072 (7)	0.060 (1)
C(18′)	−1.3607 (6)	0.5878 (2)	−0.6037 (8)	0.066 (1)
C(19′)	−1.3044 (9)	0.1972 (2)	−0.6353 (9)	0.082 (2)

Table 2. *Selected geometric parameters* (Å, °)

	Molecule 1	Molecule 2	Cl, Br and I derivatives	Ts derivative
Bond distances				
C(17)—F	1.411 (6)	1.407 (7)	–	–
C(17)—C(13)	1.518 (7)	1.514 (7)	1.523	1.528
C(13)—C(14)	1.570 (6)	1.558 (7)	1.558	1.564
C(14)—C(15)	1.566 (8)	1.543 (7)	1.563	1.552
C(15)—C(16)	1.469 (8)	1.440 (9)	1.456	1.462
C(16)—N	1.117 (8)	1.149 (9)	1.134	1.147
C(3)—O	1.388 (6)	1.372 (6)	1.376	1.376
C(19)—O	1.417 (7)	1.420 (7)	1.417	1.437
Bond angles				
C(13)—C(17)—F	109.1 (4)	110.6 (4)	115.9	109.0
C(12)—C(13)—C(17)	108.1 (4)	107.7 (4)	108.8	108.1
C(14)—C(13)—C(17)	108.3 (4)	108.4 (4)	112.7	111.2
C(13)—C(14)—C(15)	110.5 (4)	113.7 (4)	113.7	113.1
C(14)—C(15)—C(16)	111.7 (5)	117.0 (5)	115.0	113.9
C(15)—C(16)—N	179.2 (7)	176.0 (7)	178.2	178.3
C(2)—C(3)—O	123.1 (4)	124.7 (4)	116.3	124.1
C(4)—C(3)—O	116.0 (4)	115.0 (4)	124.2	116.8
Torsion angles				
C(12)—C(13)—C(17)—F	−60.1 (5)	−56.1 (5)	62.6	58.7
C(14)—C(13)—C(17)—F	−177.7 (4)	−172.9 (4)	−57.6	−61.0
C(17)—C(13)—C(14)—C(15)	−67.5 (5)	−63.0 (5)	−54.8	−59.7
C(13)—C(14)—C(15)—C(16)	140.5 (5)	−84.8 (6)	−74.7	−82.9
C(8)—C(14)—C(15)—C(16)	−93.6 (5)	44.0 (6)	53.6	45.1
C(2)—C(3)—O—C(19)	−6.2 (7)	−4.2 (7)	1.8	179.5

Table 3. *Ring-puckering coordinates and asymmetry parameters* (Å, °)

	Q	φ	θ	C_s	C_2
Ring *B*, molecule 1	0.520 (5)	340.1 (7)	130.7 (6)	–	9.2 [C(7)—C(8)]
Ring *B*, molecule 2	0.528 (5)	322.3 (7)	129.9 (6)	–	7.9 [C(7)—C(8)]
Ring *C*, molecule 1	0.569 (6)	274 (7)	175.5 (6)	5.8 [C(8)]	6.9 [C(8)—C(9)]
				3.8 [C(9)]	1.4 [C(9)—C(11)]
				2.6 [C(11)]	5.7 [C(11)—C(12)]
Ring *C*, molecule 2	0.589 (6)	217 (5)	173.1 (6)	5.3 [C(8)]	3.0 [C(8)—C(9)]
				2.1 [C(9)]	4.6 [C(9)—C(11)]
				7.4 [C(11)]	5.3 [C(11)—C(12)]

H atoms were generated and refined as riding groups (overall isotropic displacement parameters were refined for different CH types). Program used to solve structure: *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *CSU* (Vicković, 1988).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271–383.

Petrović, J. A., Pejanović, V. M., Miljković, D. A. & Hranisavljević, J. T. (1990). *Steroids*, **55**, 276–278.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Stanković, S., Petrović, J., Miljković, D., Pejanović, V., Kovačevic, R., Stefanović, A. & Bruvo, M. (1992). *Acta Cryst.* C**48**, 1248–1252.

Stanković, S., Stefanović, A., Bruvo, M. & Altomare, A. (1992). *Acta Cryst.* C**48**, 2082–2085.

Vicković, I. (1988). *CSU. Crystal Structure Utility Program.* Univ. of Zagreb, Croatia.

Acta Cryst. (1994). C**50**, 1747–1749

5-Acetyl[2.2]paracyclophane

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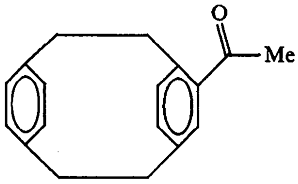
(*Received* 26 *August* 1993; *accepted* 25 *March* 1994)

Abstract

The title compound 5-acetyltricyclo[8.2.2.2^{4,7}]hexadeca-4,6,10,12,13,15-hexaene, $C_{18}H_{18}O$, is the first example of a mono-π-substituted [2.2]paracyclophane to be structurally characterized. The average bending angles are α = 13.2 and β = 9.9°. The distance between the 'bottoms' of the practically parallel boat-like benzene nuclei is 3.098 (2) Å. The π conjugation between the acetyl group and the substituted benzene cycle is negligible (rotation angle *ca* 45°) because of steric hindrance.

Comment

In order to study the influence of substituents on the degree of steric strain in [2.2]paracyclophanes (Lindeman, Struchkov & Guryshev, 1986; Lindeman, Maleev, Struchkov & Guryshev, 1990) the structure of 5-acetyl-[2.2]paracyclophane, (I), has been studied.



(I)

The benzene nuclei in molecule (I) (Fig. 1) have a boat conformation with bending angles, α, in the range 12.4–14.4 (1)° (average 13.2°). The C_{sp^2}—C_{sp^3} bonds are inclined to the adjacent triangular flaps of the benzene nuclei, exhibiting typical paracyclophane distortion with β angles in the range 8.7–10.9 (2)° (average 9.9°).

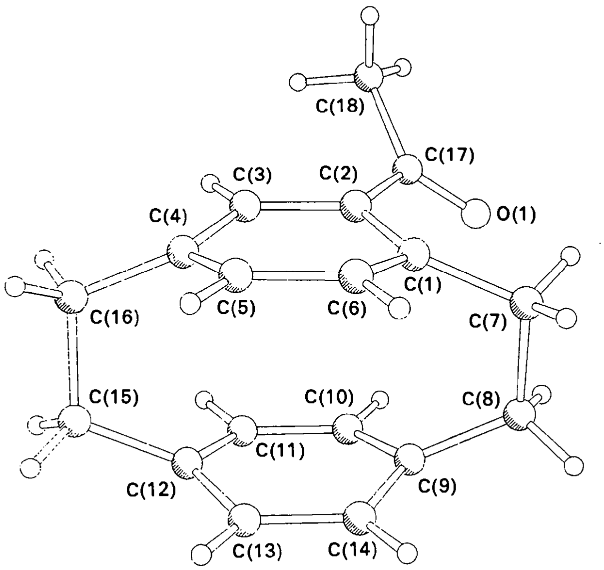


Fig. 1. Perspective view of structure (I) showing the labelling of the non-H atoms.

The C_{sp^3}—C_{sp^3} bond lengths are increased by the distortion to 1.585 (2) Å (in the bridge nearest to the acetyl substituent) and 1.574 (2) Å; the respective twist angles are −12.8 (2) and −12.3 (2)°. The same sign for both twist angles results in a relative swinging of the benzene rings by *ca* 3.5° (Fig. 2), while both 'bottom' planes are practically parallel [dihedral angle 0.2 (1)°] and located at a distance of 3.098 (2) Å from each other.

There is little conjugation between the benzene ring and the acetyl group bonded to it and the dihedral angle formed by the acetyl plane and the 'bottom' plane of the ring is 44.9 (1)°. The carbonyl atom C(17) is displaced by 0.047 (2) Å from this plane in the *exo* direction and this results in a short intramolecular O(1)···H(7*A*) distance of 2.42 (2) Å.

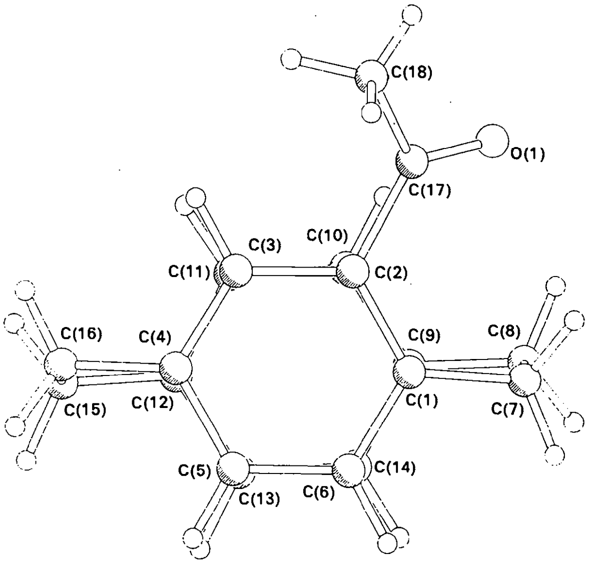


Fig. 2. Orthogonal projection of structure (I) onto the plane parallel to the 'bottoms' of the benzene nuclei showing the relative 'twist' of the benzene cycles.

Experimental

Crystal data

$C_{18}H_{18}O$
$M_r = 250.32$
Monoclinic
$P2_1/c$
$a = 14.724\ (3)$ Å
$b = 7.621\ (2)$ Å
$c = 11.844\ (2)$ Å
$\beta = 95.17\ (3)°$
$V = 1323.6\ (5)$ Å^3
$Z = 4$
$D_x = 1.256$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 24 reflections
$\theta = 11–12°$
$\mu = 0.076$ mm^{-1}
$T = 153\ (2)$ K
Well formed prisms
0.5 × 0.3 × 0.2 mm
Colourless

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3892 measured reflections
3771 independent reflections
3343 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0154$
$\theta_{\rm max} = 33.04°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 11$
$l = -18 \rightarrow 16$
2 standard reflections monitored every 98 reflections intensity variation: 2.5%

Refinement

Refinement on F^2
$R(F) = 0.0512$
$wR(F^2) = 0.1668$
$S = 1.057$
3768 reflections
244 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0969P)^2 + 0.2841P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.147$
$\Delta\rho_{\rm max} = 0.400$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.178$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.68273 (8)	0.15250 (14)	0.65165 (10)	0.0274 (2)
C2	0.65981 (7)	0.05648 (14)	0.74675 (9)	0.0259 (2)
C3	0.72487 (8)	0.03463 (15)	0.83931 (9)	0.0267 (2)
C4	0.81175 (7)	0.1071 (2)	0.84020 (9)	0.0280 (2)
C5	0.82361 (8)	0.2367 (2)	0.75984 (11)	0.0310 (2)
C6	0.76004 (8)	0.25912 (14)	0.66755 (10)	0.0304 (2)
C7	0.63974 (10)	0.1257 (2)	0.53202 (11)	0.0379 (3)
C8	0.67785 (13)	−0.0432 (2)	0.47417 (13)	0.0496 (4)
C9	0.76203 (10)	−0.1165 (2)	0.53935 (11)	0.0370 (3)
C10	0.75468 (10)	−0.2424 (2)	0.62424 (12)	0.0370 (3)
C11	0.82170 (10)	−0.2579 (2)	0.71410 (11)	0.0361 (3)
C12	0.89799 (9)	−0.1487 (2)	0.72201 (11)	0.0349 (3)
C13	0.91427 (10)	−0.0547 (2)	0.62481 (12)	0.0376 (3)
C14	0.84738 (11)	−0.0386 (2)	0.53487 (11)	0.0390 (3)
C15	0.94745 (11)	−0.1035 (3)	0.83575 (14)	0.0484 (4)
C16	0.89244 (9)	0.0251 (2)	0.90810 (12)	0.0378 (3)
C17	0.57123 (9)	−0.0395 (2)	0.74588 (12)	0.0356 (3)
C18	0.52467 (11)	−0.0402 (2)	0.8536 (2)	0.0460 (4)
O1	0.53722 (9)	−0.1125 (2)	0.66124 (10)	0.0608 (4)

Table 2. *Selected geometric parameters* (Å, °)

C1—C6	1.397 (2)	C7—C8	1.585 (2)
C1—C2	1.409 (2)	C9—C10	1.401 (2)
C1—C7	1.513 (2)	C10—C11	1.390 (2)
C2—C3	1.399 (2)	C11—C12	1.395 (2)
C2—C17	1.495 (2)	C12—C13	1.395 (2)
C3—C4	1.393 (2)	C12—C15	1.512 (2)
C4—C5	1.393 (2)	C13—C14	1.389 (2)
C4—C16	1.509 (2)	C15—C16	1.574 (2)
C5—C6	1.384 (2)	C17—O1	1.214 (2)
C6—C1—C2	116.42 (11)	C14—C9—C8	121.66 (14)
C6—C1—C7	118.14 (11)	C10—C9—C8	120.54 (14)
C2—C1—C7	124.64 (11)	C11—C10—C9	120.82 (13)
C3—C2—C1	119.45 (10)	C10—C11—C12	120.85 (12)
C3—C2—C17	118.84 (10)	C11—C12—C13	116.65 (13)
C1—C2—C17	121.35 (11)	C11—C12—C15	121.06 (13)
C4—C3—C2	121.48 (10)	C13—C12—C15	120.90 (14)
C3—C4—C5	116.59 (11)	C14—C13—C12	121.01 (13)
C3—C4—C16	121.22 (12)	C13—C14—C9	120.81 (12)
C5—C4—C16	121.09 (11)	C12—C15—C16	113.37 (11)
C6—C5—C4	120.62 (10)	C4—C16—C15	112.77 (11)
C5—C6—C1	121.29 (11)	O1—C17—C2	121.64 (13)
C1—C7—C8	112.33 (11)	O1—C17—C18	120.93 (13)
C9—C8—C7	112.73 (12)	C2—C17—C18	117.43 (12)
C14—C9—C10	116.54 (13)		
C3—C4—C16—C15	94.8 (2)	C8—C7—C1—C6	92.50 (15)
C4—C16—C15—C12	−12.3 (2)	C9—C10—C11—C12	−0.1 (2)
C16—C15—C12—C11	−71.7 (2)	C10—C11—C12—C13	−14.6 (2)
C10—C9—C8—C7	89.8 (2)	C11—C12—C13—C14	14.7 (2)
C9—C8—C7—C1	−12.8 (2)	C12—C13—C14—C9	−0.1 (2)
C8—C7—C1—C2	−76.9 (2)	C1—C2—C3—C4	0.6 (2)
C5—C4—C16—C15	−72.9 (2)	C2—C3—C4—C5	15.7 (2)
C16—C15—C12—C13	94.4 (2)	C3—C4—C5—C6	−15.8 (2)
C14—C9—C8—C7	−77.0 (2)	C4—C5—C6—C1	−0.4 (2)

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

References

Lindeman, S. V., Maleev, A. V., Struchkov, Yu. T. & Guryshev, V. N. (1990). *Izv. Akad. Nauk SSSR Ser. Khim.* pp. 588–590.

Lindeman, S. V., Struchkov, Yu. T. & Guryshev, V. N. (1976). *Izv. Akad. Nauk SSSR Ser. Khim.* pp. 2007–2010.

Sheldrick, G. M. (1989). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: HU1077). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1994). **C50**, 1749–1753

Molecular Structure and Electronic Properties of a 1-Sulfonylindolizine Derivative, 2-Isopropyl-1-methylsulfonylindolizine

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Abstract

For the title compound, $C_{12}H_{15}NO_2S$, both the crystallographic data and theoretical results (*ab initio* molecular-orbital calculations) indicate a stabilization of the symmetrical conformation of the sulfone group with respect to the indolizinic bicycle. The crystal packing and the topology of the frontier orbitals clearly suggest a charge-transfer process from the five-membered ring towards the six-membered ring of the indolizine for two adjacent molecules.

Comment

Derivatives of 1-sulfonylindolizines are being studied increasingly because of their importance as a new variety of *L*-type calcium-channel blocker and the recognition the potential impact of this class of compound on the treatment of ischemic heart disease and hypertension. The biochemical studies carried out up to now indeed indicate a new binding site for these molecules associated with the *L*-type calcium channel (Nokin *et al.*, 1989, 1990; Schmid, Romey, Barhanin & Lazdunski, 1989; Polster, Christophe, Van Damme, Houliche & Chatelain, 1990; Chatelain, Baufort, Meysmans & Clinet, 1990; Chatelain, Gubin, Manning & Sissman, 1991; Bois, Romey & Lazdunski, 1991; Gubin *et al.*, 1992; Gibon, Norberg, Vercauteren, Evrard & Durant, 1992; Kenny, Fraser & Spedding, 1993), in addition to

Acta Crystallographica Section C
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