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(1*RS*,4a*SR*,12a*SR*)-1-Methyl-1,2,3,4,4a,5,6,-7,12,12a-decahydrodibenzo[*a*,*d*]cyclooctene-1,4a-carbolactone

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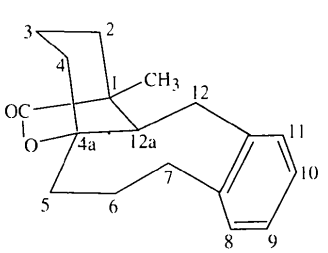
Abstract

The title compound, $C_{18}H_{22}O_2$, consists of an eight-membered carbocyclic ring fused between a six-membered aromatic and a six-membered alicyclic ring. The cyclohexane skeleton has a chair conformation, the cyclooctene ring a puckered pseudo-chair conformation and the lactone ring an envelope conformation.

Comment

Eight-membered carbocycles are of considerable current interest because of their existence in a wide variety of naturally occurring compounds showing potent pharmacological activities (Petasis & Patane, 1992; Molander & Cameron, 1993). Recently, one of us reported a highly regioselective 8-*endo*-trig-aryl cyclization leading to some decahydro[*a*,*e*]- and decahydro-[*a*,*d*]cyclooctanols (Ghosh & Ghatak, 1994). The structures of these compounds were assigned on the basis of spectroscopic data and comparison with previously reported compounds. For an unequivocal confirmation of the structures of these, and hence their congeners, we report here the X-ray crystal structure of a representative tricarbocycle, (I) (Ghosh & Ghatak, 1995). To the best of our knowledge, this is the first X-ray structure report of a compound in which an eight-membered carbocyclic ring is fused between two six-membered rings.



(I)

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Of the two six-membered rings, C1–C6 (Fig. 1) is almost planar (mean deviation from the aromatic benzene ring plane is 0.007 Å) and the other ring, C10–C15, has a chair conformation (cyclohexyl ring). The eight-membered carbocycle is puckered with a pseudo-chair conformation. In the cyclohexyl ring, the C10, C11, C13 and C14 atoms form a plane (mean deviation 0.016 Å), which makes dihedral angles of 60.8 and 41.2° with the planes constituted by atoms C10, C15, C14 and C11, C12, C13, respectively. In the eight-membered carbocycle, there are also at least two sets of four C atoms lying on planes. Atoms C16, C1, C6 and C7 form a plane (mean deviation 0.012 Å) that is almost coplanar with the aromatic ring (dihedral angle 2.1°). The other plane (mean deviation 0.010 Å) is defined by atoms C15, C16, C7 and C8, and the dihedral angle between these two planes is 67.5°. The fourth ring in the compound, *i.e.* the lactone five-membered ring, has an envelope conformation, with atoms C10, O2, C17 and C14 lying on a plane (mean deviation 0.001 Å) that makes a dihedral angle of 46.1° with the plane constituted by atoms C14, C15 and C10.

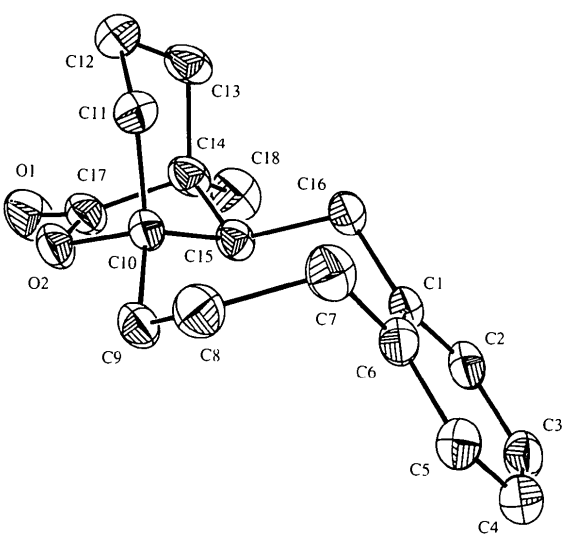

Fig. 1. The molecular structure of the title compound showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity.

The molecule has three chiral centres (atoms C10, C14 and C15 of Fig. 1), but the crystal is a racemate. The C—C single and C—C aromatic bond distances lie in the ranges 1.492 (5)–1.550 (6) and 1.368 (8)–1.404 (7) Å, respectively, with bond e.s.d.'s in the range 0.006–0.009 Å. The lactone carboxylic C—O and C=O lengths are 1.358 (6) and 1.206 (6) Å, respectively.

Experimental

The title compound was prepared according to the method of Ghosh & Ghatak (1995). Single crystals were prepared by slow evaporation of a petroleum ether solution of the

compound. The density D_m was measured by flotation in 27% aqueous NaCl solution.

Crystal data

$C_{18}H_{22}O_2$	Mo $K\alpha$ radiation
M_r = 270.4	λ = 0.71073 Å
Orthorhombic	Cell parameters from 30 reflections
Pbca	θ = 8–15°
a = 11.873 (2) Å	μ = 0.077 mm^{-1}
b = 14.322 (4) Å	T = 295 K
c = 17.541 (6) Å	Rectangular parallelepiped
V = 2983 (1) Å^3	0.34 × 0.32 × 0.28 mm
Z = 8	Colourless
D_x = 1.204 Mg m^{-3}	
D_m = 1.20 Mg m^{-3}	

Data collection

Siemens *R3m/V* diffractometer	θ_{max} = 22.5°
ω scans	h = 0 → 12
Absorption correction: none	k = 0 → 15
1965 measured reflections	l = 0 → 18
1965 independent reflections	2 standard reflections monitored every 98 reflections
1316 observed reflections [$I > \sigma(I)$]	intensity decay: 3.0%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} < 0.001$
R = 0.0708	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
wR = 0.0705	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
S = 1.09	Extinction correction: none
1316 reflections	Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)
181 parameters	
$w = 1/[\sigma^2(F) + 0.0020F^2]$	

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}
C(1)	0.1694 (4)	0.4852 (3)	0.2472 (3)	0.041 (2)
C(2)	0.0707 (4)	0.5354 (3)	0.2567 (3)	0.051 (2)
C(3)	0.0082 (4)	0.5640 (3)	0.1935 (4)	0.060 (2)
C(4)	0.0426 (5)	0.5411 (3)	0.1214 (3)	0.061 (2)
C(5)	0.1401 (5)	0.4916 (3)	0.1116 (3)	0.054 (2)
C(6)	0.2065 (4)	0.4636 (3)	0.1736 (3)	0.045 (2)
C(7)	0.3146 (4)	0.4134 (3)	0.1590 (3)	0.052 (2)
C(8)	0.3071 (4)	0.3068 (3)	0.1530 (3)	0.056 (2)
C(9)	0.2415 (4)	0.2558 (3)	0.2162 (3)	0.049 (2)
C(10)	0.2748 (4)	0.2757 (3)	0.2989 (2)	0.041 (2)
C(11)	0.4016 (4)	0.2850 (3)	0.3133 (3)	0.047 (2)
C(12)	0.4287 (4)	0.2819 (4)	0.3987 (3)	0.062 (2)
C(13)	0.3472 (5)	0.3408 (3)	0.4460 (3)	0.057 (2)
C(14)	0.2239 (4)	0.3230 (3)	0.4229 (2)	0.049 (2)
C(15)	0.2062 (4)	0.3521 (3)	0.3387 (2)	0.040 (1)
C(16)	0.2321 (4)	0.4540 (3)	0.3181 (2)	0.044 (2)
C(17)	0.2082 (5)	0.2195 (3)	0.4144 (3)	0.054 (2)
C(18)	0.1416 (5)	0.3663 (4)	0.4781 (3)	0.078 (2)
O(1)	0.1796 (3)	0.1629 (3)	0.4615 (2)	0.086 (2)
O(2)	0.2371 (3)	0.1922 (2)	0.3429 (2)	0.052 (1)

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

C(1)—C(2)	1.385 (6)	C(1)—C(6)	1.399 (7)
C(1)—C(16)	1.517 (6)	C(2)—C(3)	1.395 (8)
C(3)—C(4)	1.369 (9)	C(4)—C(5)	1.368 (8)
C(5)—C(6)	1.404 (7)	C(6)—C(7)	1.492 (7)
C(7)—C(8)	1.532 (7)	C(8)—C(9)	1.540 (7)
C(9)—C(10)	1.531 (6)	C(10)—C(11)	1.531 (7)
C(10)—C(15)	1.532 (6)	C(10)—O(2)	1.492 (5)
C(11)—C(12)	1.533 (7)	C(12)—C(13)	1.528 (7)
C(13)—C(14)	1.540 (7)	C(14)—C(15)	1.550 (6)
C(14)—C(17)	1.502 (7)	C(14)—C(18)	1.509 (7)
C(15)—C(16)	1.535 (6)	C(17)—O(1)	1.206 (6)
C(17)—O(2)	1.358 (6)		
C(2)—C(1)—C(6)	119.5 (4)	C(2)—C(1)—C(16)	118.0 (4)
C(6)—C(1)—C(16)	122.5 (4)	C(1)—C(2)—C(3)	120.4 (5)
C(2)—C(3)—C(4)	120.4 (5)	C(3)—C(4)—C(5)	119.5 (5)
C(4)—C(5)—C(6)	121.7 (5)	C(1)—C(6)—C(5)	118.4 (4)
C(1)—C(6)—C(7)	122.4 (4)	C(5)—C(6)—C(7)	119.2 (4)
C(6)—C(7)—C(8)	116.2 (4)	C(7)—C(8)—C(9)	116.9 (4)
C(8)—C(9)—C(10)	117.6 (4)	C(9)—C(10)—C(11)	115.3 (4)
C(9)—C(10)—C(15)	115.3 (4)	C(11)—C(10)—C(15)	112.6 (3)
C(9)—C(10)—O(2)	105.3 (3)	C(11)—C(10)—O(2)	106.2 (3)
C(15)—C(10)—O(2)	100.2 (3)	C(10)—C(11)—C(12)	111.4 (4)
C(11)—C(12)—C(13)	112.5 (4)	C(12)—C(13)—C(14)	111.6 (4)
C(13)—C(14)—C(15)	109.6 (4)	C(13)—C(14)—C(17)	107.9 (4)
C(15)—C(14)—C(17)	98.9 (3)	C(13)—C(14)—C(18)	112.2 (4)
C(15)—C(14)—C(18)	114.5 (4)	C(17)—C(14)—C(18)	112.9 (4)
C(10)—C(15)—C(14)	99.8 (3)	C(10)—C(15)—C(16)	117.8 (4)
C(14)—C(15)—C(16)	116.9 (3)	C(1)—C(16)—C(15)	112.0 (3)
C(14)—C(17)—O(1)	129.1 (5)	C(14)—C(17)—O(2)	110.1 (4)
O(1)—C(17)—O(2)	120.7 (4)	C(10)—O(2)—C(17)	108.9 (3)

The structure was solved by direct methods and subsequent difference Fourier syntheses, and refined by full-matrix least-squares procedures. All non-H atoms were refined anisotropically. H atoms were located on a difference Fourier map, but their positional and displacement (assigned fixed U_{iso} = 0.08 Å^2) parameters were not refined.

Data collection: *P3* (Siemens, 1982). Cell refinement: *P3*. Data reduction: *P3*. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus XP*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with packing diagrams viewed down the *a*, *b* and *c* axes, have been deposited with the IUCr (Reference: DE1005). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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