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Structure of 10,11-Dihydro-5,10-ethano-5*H*-dibenzo[a,d]cycloheptan-11-one

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Abstract. $C_{17}H_{14}O$ (1), $M_r = 234.3$, monoclinic, $P2_1/c$, $a = 9.587$ (2), $b = 11.091$ (2), $c = 11.815$ (2) Å, $\beta = 102.00$ (1)°, $V = 1228.8$ (4) Å^3, $Z = 4$, $D_x = 1.266$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 0.72$ cm^{-1}, $F(000) = 496$, $T = 193$ K, $R = 0.0387$ and $wR = 0.0411$ for 1529 reflections $[I \geq 3\sigma(I)]$. The molecule consists of two benzene rings fused on opposite sides of a cycloheptane ring with an ethano group bridging C(5) and C(10). The four-membered chain of C(5), C(13), C(12) and C(10) is nearly planar [angle C(5)—C(13)—C(12)—C(10) is 6.8 (3)°], and it forms dihedral angles of 118.0 (4) and 128.9 (4)° with the C(1) and the C(6) phenyl rings, respectively. The dihedral angle between the phenyl rings is 112.9 (4)°. The two 7-membered rings [C(11a), C(4a), C(5), C(13), C(12), C(10), C(11) and C(11a), C(4a), C(5), C(5a), C(9a), C(10), C(11)] are in boat–sofa conformations, BS11a and BS11a, respectively. Their puckering parameters [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1358] $q(2)$, $q(3)$, $\varphi(2)$, $\varphi(3)$ and the total puckering amplitude, Q, are 0.943, 0.356 Å, 185.2, 2.3°, 1.008 Å, for the former, and 0.925, 0.301 Å, 355.9, 182.0°, 0.973 Å, for the latter. The 6-membered ring [C(5), C(13), C(12), C(10), C(9a), C(5a)] is in a boat conformation, $^{5,10}B$, and it's puckering parameters $q(2)$, $q(3)$, $\varphi(2)$, and the puckering amplitude, Q, are 0.688, 0.022 Å, 175.4°, 0.689 Å, respectively. The carbonyl oxygen is involved in two close intermolecular contacts with the aromatic H(7) and H(9) atoms $[O(1)\cdots H(7), (-x, 0.5+y, 1.5-z) = 2.53$ (2) Å,

$O(1)\cdots C(7) = 3.36$ (1) Å, $O(1)\cdots H(7)$—$C(7) = 117$ (1)°; $O(1)\cdots H(9)$, $(-x, 1-y, 1-z) = 2.62$ (2) Å, $O(1)\cdots C(9) = 3.61$ (1) Å, $O(1)\cdots H(9)$—$C(9) = 141$ (1)°].

Experimental. A 50 ml round-bottom flask equipped with a reflux condenser and a magnetic stirring bar was charged with 973 mg (4.1 mmol) of 10,11-dihydro-5,10-ethano-5*H*-dibenzo[a,d]cyclohepten-11-ol, 788 mg (4.4 mmol) of *N*-bromosuccinimide, 12 mg of benzoyl peroxide and 20 ml of carbon tetrachloride. The mixture was stirred and irradiated with a 150 W unfrosted incandescent bulb until the *N*-bromosuccinimide was completely consumed (about 45 min). The mixture was washed with two 10 ml portions of water, one 10 ml portion of saturated sodium hydrogencarbonate solution, and two 10 ml portions of 0.05 *M* sodium thiosulfate solution. The organic layer was stripped of solvent and the residue dissolved in chloroform; that solution was eluted through a short column of activated alumina. The eluate was concentrated, affording a bright-yellow oil that crystallized on standing to yield 855 mg (90% of theoretical) of 10,11-dihydro-5,10-ethano-5*H*-dibenzo[a,d]cyclohepten-11-one, m.p. 383–386 K. The product was identified by ^{1}HMR and by ^{13}CMR. The crystal used in the X-ray analysis was selected from a few excellent specimens produced in a single crystallization from a solution of the product in a 10:1 mixture of hexanes and chloroform, and was a pale-yellow prism of dimen-

sions $0.12 \times 0.18 \times 0.22$ mm. Data were collected on a Siemens $R3m/V$ diffractometer equipped with a graphite monochromator utilizing Mo $K\alpha$ radiation $(\lambda = 0.71073$ Å). 50 reflections with $21.08 \le 2\theta \le 27.86°$ were used to refine the cell parameters. 5829 reflections were collected using the ω-scan method (h, $0 \rightarrow 12$; k, $-14 \rightarrow 14$; l, $-15 \rightarrow 15$), 2815 unique reflections, $R_{int} = 0.0287$; 2θ range $3 \rightarrow 55°$, $1.0°$ ω scan at 6–$12°$ min^{-1}, depending upon intensity. Four reflections $(115, 023, 12\overline{3}, \overline{2}\overline{2}0)$ were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.02\%$). Absorption corrections were not applied owing to the crystal size and the small value of the absorption coefficient $(\mu = 0.72$ cm$^{-1})$.
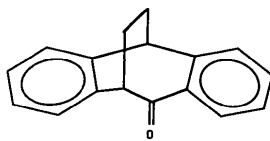
(1)

The structure was solved by direct methods in *SHELXTL-Plus* (Sheldrick, 1987) from which the locations of all non-H atoms were obtained. The structure was refined (*SHELX76*; Sheldrick, 1976) using full-matrix least squares and the positions of all H atoms were determined from a difference Fourier map. The non-H atoms were treated aniso-tropically, whereas the H atoms were refined with isotropic thermal parameters. 220 parameters were refined and $\sum w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0.5\,kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, $I_{intensity} = (I_{peak} - I_{background})$ (scan rate), and $\sigma(I) = (I_{peak} + I_{background})^{1/2}$ (scan rate), k is the correction due to decay and Lp effects, 0.02 is a factor used to downweight intense reflections and to account for instrument instability. An extinction correction $\chi = 0.0081$ (7) {where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$} was also applied (Sheldrick, 1987). Final $R = 0.0387$, $wR = 0.0411$ ($R_{all} = 0.0828$, $wR_{all} = 0.0582$) for 1529
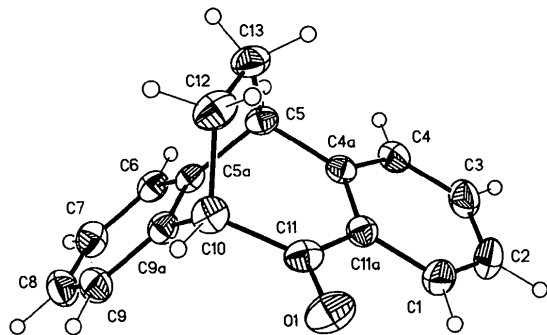

Fig. 1. Molecular structure of (1), with 50% probability ellipsoids, showing the atom-numbering scheme.

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

	x	y	z	U_{eq}*
O(1)	2625 (2)	4459 (1)	5749 (1)	67 (1)
C(1)	4282 (2)	3496 (2)	7738 (2)	54 (1)
C(2)	5150 (2)	2991 (2)	8696 (2)	66 (1)
C(3)	4940 (2)	1811 (2)	8978 (2)	63 (1)
C(4)	3863 (2)	1144 (2)	8312 (2)	49 (1)
C(4a)	2969 (2)	1628 (2)	7343 (1)	38 (1)
C(5)	1794 (2)	848 (2)	6662 (2)	42 (1)
C(5a)	359 (2)	1386 (2)	6700 (1)	37 (1)
C(6)	−553 (2)	923 (2)	7361 (2)	45 (1)
C(7)	−1840 (2)	1491 (2)	7364 (2)	54 (1)
C(8)	−2221 (2)	2503 (2)	6704 (2)	56 (1)
C(9)	−1330 (2)	2971 (2)	6030 (2)	51 (1)
C(9a)	−32 (2)	2416 (2)	6036 (2)	41 (1)
C(10)	993 (2)	2851 (2)	5310 (2)	46 (1)
C(11)	2300 (2)	3455 (2)	6023 (2)	44 (1)
C(11a)	3188 (2)	2829 (2)	7043 (2)	41 (1)
C(12)	1431 (3)	1765 (2)	4632 (2)	57 (1)
C(13)	1965 (3)	690 (2)	5394 (2)	55 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°) *of the non-*H *atoms*

O(1)—C(11)	1.218 (2)	C(1)—C(2)	1.377 (7)
C(1)—C(11a)	1.401 (3)	C(2)—C(3)	1.376 (4)
C(3)—C(4)	1.376 (3)	C(4)—C(4a)	1.388 (2)
C(4a)—C(5)	1.512 (2)	C(4a)—C(11a)	1.406 (3)
C(5)—C(5a)	1.509 (3)	C(5)—C(13)	1.551 (3)
C(5a)—C(6)	1.387 (3)	C(5a)—C(9a)	1.392 (2)
C(6)—C(7)	1.386 (3)	C(7)—C(8)	1.372 (3)
C(8)—C(9)	1.385 (3)	C(9)—C(9a)	1.387 (3)
C(9a)—C(10)	1.512 (3)	C(10)—C(11)	1.513 (3)
C(10)—C(12)	1.551 (3)	C(11)—C(11a)	1.494 (2)
C(12)—C(13)	1.519 (3)		
C(2)—C(1)—C(11a)	121.2 (2)	C(1)—C(2)—C(3)	119.5 (2)
C(2)—C(3)—C(4)	120.2 (2)	C(3)—C(4)—C(4a)	121.7 (2)
C(4)—C(4a)—C(5)	118.8 (2)	C(4)—C(4a)—C(11a)	118.4 (2)
C(5)—C(4a)—C(11a)	122.8 (1)	C(4a)—C(5)—C(5a)	110.0 (2)
C(4a)—C(5)—C(13)	111.2 (2)	C(5a)—C(5)—C(13)	110.7 (2)
C(5)—C(5a)—C(6)	123.8 (2)	C(5)—C(5a)—C(9a)	116.7 (2)
C(6)—C(5a)—C(9a)	119.6 (2)	C(5a)—C(6)—C(7)	119.9 (2)
C(6)—C(7)—C(8)	120.2 (2)	C(7)—C(8)—C(9)	120.7 (2)
C(8)—C(9)—C(9a)	119.4 (2)	C(5a)—C(9a)—C(9)	120.2 (2)
C(5a)—C(9a)—C(10)	116.8 (2)	C(9)—C(9a)—C(10)	122.9 (2)
C(9a)—C(10)—C(11)	112.7 (2)	C(9a)—C(10)—C(12)	108.8 (2)
C(11)—C(10)—C(12)	110.5 (2)	O(1)—C(11)—C(10)	119.0 (2)
O(1)—C(11)—C(11a)	120.3 (2)	C(10)—C(11)—C(11a)	120.7 (2)
C(1)—C(11a)—C(4a)	119.1 (2)	C(1)—C(11a)—C(11)	117.3 (2)
C(4a)—C(11a)—C(11)	123.6 (2)	C(10)—C(12)—C(13)	113.5 (2)
C(5)—C(13)—C(12)	113.2 (2)		

reflections having $I \ge 3\sigma(I)$, and goodness-of-fit = 1.22. Maximum $\Delta/\sigma = 0.003$ in the final refinement cycle and the minimum and maximum peaks in the ΔF map were -0.15 and 0.22 e Å^{-3}, respectively. The linear absorption coefficient was calculated from values from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with anomalous-dispersion corrections from Cromer & Liberman (1970), while those of H atoms were from Stewart, Davidson & Simpson

(1965). The positional parameters and the equivalent isotropic thermal parameters of the non-H atoms are listed in Table 1;* bond lengths and angles are listed in Table 2. The thermal ellipsoid drawing (SHELXTL-Plus; Sheldrick, 1987) of the molecule with the atom-labelling scheme is given in Fig. 1. The ring puckering parameters (Cremer & Pople, 1975; Boeyens, 1978; Boessenkool & Boeyens, 1980) were calculated using PLATON (Spek, 1990).

Related literature. Abboud, Simonsen, Tyllick & Roberts (1988, 1991); Cioranescu, Banciu, Jelescu, Rentzea, Elian & Nenitzescu (1969a,b); Cristol & Noreen (1976).

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* Lists of structure factors, anisotropic thermal parameters, crystallographic data, bond lengths, bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53534 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 10,11-Dihydro-5,10-ethano-5H-dibenzo[a,d]cycloheptene

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Abstract. $C_{17}H_{16}$ (1), $M_r = 220.3$, monoclinic, $P2_1/n$, $a = 8.465$ (1), $b = 6.509$ (2), $c = 21.305$ (8) Å, $\beta = 93.44$ (2)°, $V = 1171.6$ (5) Å^3, $Z = 4$, $D_x = 1.249$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0.71073$ Å), $\mu = 0.65$ cm^{-1}, $F(000) = 472$, $T = 193$ K, $R = 0.0461$ and $wR = 0.0679$ for 2200 reflections [$I \geq 3\sigma(I)$]. The molecule consists of two benzene rings fused on opposite sides of a cycloheptane ring with an ethano group bridging C(5) and C(10). The 4-membered chain of C(5), C(13), C(12) and C(10) is nearly planar [angle C(5)—C(13)—C(12)—C(10) is −5.7 (2)°], but is considerably different from its counterpart in a similar compound, 10,11-dihydro-5,10-ethano-5H-dibenzo[a,d]cyclohepten-11-one (2), studied in our laboratory, which has an angle of 6.8 (3)°. The chain forms dihedral angles of 114.6 (4) and 127.4 (4)° with the C(1) and the C(6) benzene rings, respectively. The dihedral angle between the phenyl rings is 118.0 (3)°. These three dihedral angles differ significantly from their counterparts in (2) where values of 118.0 (4), 128.9 (4) and 112.9 (4)° were observed. The ring conformations are the same as in (2). The two 7-membered rings [C(11a), C(4a), C(5), C(13), C(12), C(10), C(11) and C(11a), C(4a), C(5), C(5a), C(9a), C(10), C(11)] are in boat–sofa conformations, BS11a and BS11a, respectively, with Cremer & Pople [J. Am. Chem. Soc. (1975), **97**, 1354–1358] puckering parameters $q(2)$, $q(3)$, $\varphi(2)$, $\varphi(3)$ and the total puckering amplitude, Q, of 0.953, 0.331 Å, 177.8, 355.7°, 1.009 Å, for the former, and 0.888, 0.332 Å, 0.4, 177.6°, 0.948 Å, for the latter. The 6-membered ring [C(5), C(13), C(12), C(10), C(9a), C(5a)] is in a boat conformation, $^{5,10}B$, and its puckering parameters $q(2)$, $q(3)$, $\varphi(2)$, and the puckering amplitude, Q, are 0.695, −0.032 Å, 182.5°, 0.695 Å.