(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-5*H*-dibenzo[a,d]cycloheptene

BY K. A. ABBOUD, S. H. SIMONSEN, C. S. TYLLICK AND R. M. ROBERTS

Department of Chemistry, The University of Texas at Austin, Austin, TX 78712, USA

Abstract. $C_{17}H_{16}$ (1), $M_r = 220\cdot3$, monoclinic, $P2_1/n$, $a = 8\cdot465$ (1), $b = 6\cdot509$ (2), $c = 21\cdot305$ (8) Å, $\beta = 93\cdot44$ (2)°, $V = 1171\cdot6$ (5) Å^3, $Z = 4$, $D_x = 1\cdot249$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0\cdot71073$ Å), $\mu = 0\cdot65$ cm^{-1}, $F(000) = 472$, $T = 193$ K, $R = 0\cdot0461$ and $wR = 0\cdot0679$ 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 $-5\cdot7$ (2)°], but is considerably different from its counterpart in a similar compound, 10,11-dihydro-5,10-ethano-5*H*-dibenzo[a,d]cyclohepten-11-one (2), studied in our laboratory, which has an angle of $6\cdot8$ (3)°. The chain forms dihedral angles of $114\cdot6$ (4) and $127\cdot4$ (4)° with the C(1) and the C(6) benzene rings, respectively. The dihedral angle between the phenyl rings is $118\cdot0$ (3)°. These three dihedral angles differ significantly from their counterparts in (2) where values of $118\cdot0$ (4), $128\cdot9$ (4) and $112\cdot9$ (4)° were observed. The ring conformations are the same as in (2). The two 7-membered rings [C(11*a*), C(4*a*), C(5), C(13), C(12), C(10), C(11) and C(11*a*), C(4*a*), C(5), C(5*a*), C(9*a*), C(10), C(11)] are in boat–sofa conformations, BS$\overline{11}a$ and BS11*a*, 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 $0\cdot953$, $0\cdot331$ Å, $177\cdot8$, $355\cdot7$°, $1\cdot009$ Å, for the former, and $0\cdot888$, $0\cdot332$ Å, $0\cdot4$, $177\cdot6$°, $0\cdot948$ Å, for the latter. The 6-membered ring [C(5), C(13), C(12), C(10), C(9*a*), C(5*a*)] is in a boat conformation, $^{5,10}B$, and its puckering parameters $q(2)$, $q(3)$, $\varphi(2)$, and the puckering amplitude, Q, are $0\cdot695$, $-0\cdot032$ Å, $182\cdot5$°, $0\cdot695$ Å.

Experimental. The title compound was prepared according to a published procedure (Low & Roberts, 1973). Crystals suitable for X-ray investigation were obtained after two recrystallizations of the pure product, m.p. 341–343 K, from hexanes. A colorless plate of dimensions $0.13 \times 0.20 \times 0.33$ mm was selected for data collection. 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 $27.92 \le 2\theta \le 33.93°$ were used to refine the cell parameters. 5754 reflections were collected using the ω-scan method (h, $0 \rightarrow 11$; k, $-9 \rightarrow 9$; l, $-27 \rightarrow 27$), 2695 unique reflections, $R_{int} = 0.0255$; 2θ range $3 \rightarrow 55°$, $1.2°$ ω scan at 4.5–$9°$ min^{-1}, depending upon intensity. Four reflections ($\overline{4}00$, 122, 210, $00\overline{6}$) were measured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.05\%$). Absorption corrections were not applied due to the crystal size and the small value of the absorption coefficient ($\mu = 0.65$ cm^{-1}).
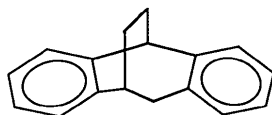
(1)

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 anisotropically, whereas the H atoms were refined with isotropic thermal parameters. 219 parameters were refined and $\Sigma w(|F_o| - |F_c|)^2$ was minimized; $w = 1/(\sigma|F_o|)^2$, $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$, $I_{intensity} = (I_{peak} - I_{background})$ (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 $\chi = 0.0022$ (7) {where $F^* = F[1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$} was also applied (Sheldrick, 1987). Final $R = 0.0461$, $wR = 0.0679$ ($R_{all} = 0.0558$, $wR_{all} = 0.0705$) for 2200 reflections having $I \ge 3\sigma(I)$, and goodness-of-fit = 2.55. Maximum $\Delta/\sigma = 0.001$ in the final refinement cycle and the min. and max. peaks in the ΔF map were -0.20 and 0.47 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

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

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

	x	y	z	U_{eq}
C(1)	4706 (2)	7538 (3)	2118 (1)	40 (1)
C(2)	5268 (2)	6183 (3)	1695 (1)	44 (1)
C(3)	5917 (2)	4369 (3)	1903 (1)	42 (1)
C(4)	5990 (2)	3914 (3)	2535 (1)	34 (1)
C(4a)	5376 (2)	5238 (2)	2971 (1)	27 (1)
C(5)	5472 (2)	4609 (2)	3658 (1)	27 (1)
C(5a)	6404 (2)	6120 (2)	4061 (1)	25 (1)
C(6)	7896 (2)	5704 (2)	4332 (1)	31 (1)
C(7)	8689 (2)	7166 (3)	4703 (1)	34 (1)
C(8)	8000 (2)	9039 (3)	4802 (1)	32 (1)
C(9)	6511 (2)	9457 (2)	4531 (1)	30 (1)
C(9a)	5699 (2)	8008 (2)	4165 (1)	26 (1)
C(10)	4052 (2)	8316 (3)	3870 (1)	32 (1)
C(11)	4073 (2)	8734 (3)	3169 (1)	39 (1)
C(11a)	4727 (2)	7102 (2)	2758 (1)	31 (1)
C(12)	3004 (2)	6428 (3)	4002 (1)	38 (1)
C(13)	3831 (2)	4365 (3)	3919 (1)	33 (1)

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

|			
C(1)—C(2)	1.366 (3)	C(1)—C(11a)	1.393 (2)
C(2)—C(3)	1.365 (3)	C(3)—C(4)	1.378 (2)
C(4)—C(4a)	1.390 (2)	C(4a)—C(5)	1.517 (2)
C(4a)—C(11a)	1.396 (2)	C(5)—C(5a)	1.499 (2)
C(5)—C(13)	1.535 (2)	C(5a)—C(6)	1.384 (2)
C(5a)—C(9a)	1.389 (2)	C(6)—C(7)	1.383 (2)
C(7)—C(8)	1.373 (2)	C(8)—C(9)	1.382 (2)
C(9)—C(9a)	1.380 (2)	C(9a)—C(10)	1.508 (2)
C(10)—C(11)	1.518 (2)	C(10)—C(12)	1.552 (2)
C(11)—C(11a)	1.503 (2)	C(12)—C(13)	1.530 (2)
C(2)—C(1)—C(11a)	122.0 (2)	C(1)—C(2)—C(3)	119.6 (2)
C(2)—C(3)—C(4)	119.7 (2)	C(3)—C(4)—C(4a)	121.6 (2)
C(4)—C(4a)—C(5)	118.4 (1)	C(4)—C(4a)—C(11a)	118.5 (1)
C(5)—C(4a)—C(11a)	123.0 (1)	C(4a)—C(5)—C(5a)	111.8 (1)
C(4a)—C(5)—C(13)	112.3 (1)	C(5a)—C(5)—C(13)	108.5 (1)
C(5)—C(5a)—C(6)	123.1 (1)	C(5)—C(5a)—C(9a)	117.1 (1)
C(6)—C(5a)—C(9a)	119.8 (1)	C(5a)—C(6)—C(7)	120.2 (1)
C(6)—C(7)—C(8)	120.2 (1)	C(7)—C(8)—C(9)	119.7 (1)
C(8)—C(9)—C(9a)	120.8 (1)	C(5a)—C(9a)—C(9)	119.4 (1)
C(5a)—C(9a)—C(10)	116.5 (1)	C(9)—C(9a)—C(10)	124.2 (1)
C(9a)—C(10)—C(11)	111.6 (1)	C(9a)—C(10)—C(12)	110.1 (1)
C(11)—C(10)—C(12)	111.2 (1)	C(10)—C(11)—C(11a)	118.2 (1)
C(1)—C(11a)—C(4a)	118.5 (1)	C(1)—C(11a)—C(11)	116.3 (1)
C(4a)—C(11a)—C(11)	125.2 (1)	C(10)—C(12)—C(13)	113.8 (1)
C(5)—C(13)—C(12)	112.4 (1)		
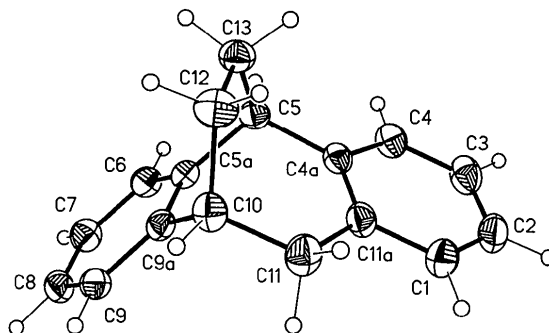
Fig. 1. Molecular structure of (1), with 50% probability ellipsoids, showing the atom-numbering scheme.

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 given 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, 1985; Boeyens, 1978; Boessenkool & Boeyens, 1980) were calculated using program *PLATON* (Spek, 1990).

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

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* Tables of the crystallographic data, anisotropic thermal parameters, H-atom positional parameters and bond lengths and angles, a packing diagram and the structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53541 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Amino-4-methyl-5-phenylthiophene-3-carboxamide

By P. S. Joseph*

Department of Physics, Bharathidasan University, Tiruchirapalli 620 024, India

S. Selladurai

Department of Physics, Anna University, Madras 600 025, India

S. Kannan

Department of Chemistry, IGNOU, New Delhi 110 030, India

AND V. Parthasarathi†

Department of Physics, Bharathidasan University, Tiruchirapalli 620 024, India

Abstract. $C_{12}H_{12}N_2OS$, $M_r = 232 \cdot 2$, triclinic, $P\bar{1}$, $a = 7 \cdot 690$ (3), $b = 7 \cdot 898$ (3), $c = 9 \cdot 923$ (2) Å, $\alpha = 67 \cdot 032$ (2), $\beta = 83 \cdot 92$ (2), $\gamma = 78 \cdot 885$ (2)°, $V = 544 \cdot 1$ (3) Å^3, $Z = 2$, $D_x = 1 \cdot 418$ g cm^{-3}, λ(Cu $K\alpha$) = $1 \cdot 5418$ Å, $\mu = 23 \cdot 81$ cm^{-1}, $F(000) = 244$, $T = 295$ K, $R = 0 \cdot 05$ for 2002 observed reflections $[I > 3\sigma(I)]$. Molecular geometry is within the normal limits. The thiophene ring is planar [max. deviation: C(2), $0 \cdot 003$ (2) Å] as is the phenyl ring [max. deviation: C(56), $0 \cdot 010$ (2) Å]. The amino, carboxamido, methyl and phenyl groups are attached preferentially to

* Present address: Department of Physics, Periyar E. V. R. College, Tiruchirapalli 620 023, India.
† To whom correspondence should be addressed.