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Conformation and Structure of 9-Propylanthracene

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Abstract. $C_{17}H_{16}$, $M_r = 220\cdot23$, orthorhombic, *Pbca*, $a = 7\cdot816\,(2)$, $b = 20\cdot455\,(4)$, $c = 16\cdot204\,(4)$ Å, $V = 2591$ Å^3, $Z = 8$, $D_x = 1\cdot13$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot6$ cm^{-1}, $F(000) = 944$, $T = 293$ K, $wR = 0\cdot057$ for 1167 unique observed $[F > 3\sigma(F)]$ reflections. The propyl side chain is perpendicular to the ring system and itself adopts an extended conformation. There is a slight folding of the molecule along the C(9)—C(10) axis, the angle between the two outer aromatic ring planes being 3·6 (5)°. There are no unusually close intermolecular contacts.

Introduction. The conformation about a bond between an aromatic sp^2 carbon and an sp^3 carbon (*e.g.* to a saturated carbon chain) depends on the nature of the aromatic ring. In ethylbenzene, the simplest molecule of this type, the existence of eclipsed (coplanar), skew and perpendicular conformers has been deduced from the experimental data. The energy barrier between the different conformers is very low (< 11·7 kJ mol^{-1}; Schrumpf, unpublished results). The situation is probably analogous to that in the homologues with longer straight-chain substituents. 9-Propylanthracene might be considered as an *ortho*-disubstituted benzene displaying steric interactions between the *peri* H atoms and the chain in ecliptic or skew conformations. It appeared interesting to study the conformation of this molecule in the crystal and to investigate whether the

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deformations of the aromatic ring exceed those present in 9-methylanthracene, the crystal structure of which is known (Cox & Sim, 1979). We here report the X-ray study of 9-propylanthracene.

Experimental. 9-Propylanthracene was synthesized according to the literature (Lalande & Calas, 1959). Pale-yellow crystals (m.p. 342 K) were obtained from acetone/water. A prism 0·7 × 0·3 × 0·15 mm was used to record 3700 profile-fitted intensities (Clegg, 1981) on a Stoe–Siemens four-circle diffractometer ($2\theta_{\max}$ 50°, octant $-h+k+l$ and some $+h$ equivalents). Merging equivalents gave 2278 unique reflections ($R_{\rm int}$ 0·049), of which 1167 with $F > 3\sigma(F)$ were used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Cell constants were refined from 2θ values of 60 reflections in the range 20–23°. No correction for absorption. No intensity variation of standard reflections.

The structure was solved by routine direct methods and refined anisotropically on F to R 0·101, wR 0·057. The high R value is caused by the weakly diffracting crystal. H atoms were included using a riding model with C—H 0·96 Å, H—C—H 109·5°, $U(\mathrm{H}) = 1{\cdot}2\,U_{\rm eq}(\mathrm{C})$. The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}00012F^2$. 157 parameters; max. Δ/σ 0·04; max. $\Delta\rho$ ±0·025 e Å^{-3}, S 1·6. Atomic scattering factors from *SHELXTL*.

Discussion. Final atomic coordinates and derived bond lengths and angles are presented in Tables 1 and 2.* The molecule is illustrated in Fig. 1, the crystal packing in Fig. 2. The anisotropic thermal parameters were analysed in terms of a rigid-body libration (Schomaker & Trueblood, 1968), giving $R_{\rm lib}$ 0·084, indicating that the thermal motion can be reasonably approximated in such a manner. However, uncorrected bond lengths are used in the discussion.

The conformation of the propyl side chain about C(9)—C(91) is perpendicular, with torsion angles C(9a)—C(9)—C(91)—C(92) 89·8 (5), C(8a)—C(9)—C(91)—C(92) −88·1 (6)°. The propyl group itself displays an *anti* (extended) conformation about C(91)—C(92) [178·7 (5)°]. Clearly, the interactions between H(1), H(8) and H(91*a*), H(91*b*) are minimized in this geometry. This is also obvious from the structure of the aromatic skeleton.

The bond angles and bond lengths in the region about C(9) are larger than in anthracene (Cruickshank, 1956), but smaller in the region of C(10). The

Table 1. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	$U_{\rm eq}$*
C(1)	4913 (6)	3096 (2)	3331 (3)	68 (2)
C(2)	5818 (6)	2599 (2)	2992 (3)	81 (2)
C(3)	7158 (6)	2295 (2)	3435 (3)	80 (2)
C(4)	7551 (6)	2500 (2)	4197 (3)	71 (2)
C(4a)	6635 (5)	3018 (2)	4580 (3)	52 (2)
C(5)	6486 (6)	3880 (2)	6599 (3)	71 (2)
C(6)	5544 (6)	4338 (2)	7001 (3)	81 (2)
C(7)	4187 (7)	4653 (2)	6581 (4)	80 (2)
C(8)	3804 (5)	4504 (2)	5791 (3)	67 (2)
C(8a)	4733 (5)	4020 (2)	5345 (3)	51 (2)
C(9)	4346 (4)	3841 (2)	4533 (3)	49 (2)
C(9a)	5270 (5)	3332 (2)	4146 (3)	52 (2)
C(10)	7039 (5)	3211 (2)	5373 (3)	59 (2)
C(10a)	6120 (5)	3702 (2)	5768 (2)	53 (2)
C(91)	2895 (5)	4175 (2)	4073 (3)	63 (2)
C(92)	1153 (5)	3853 (2)	4202 (3)	73 (2)
C(93)	−250 (5)	4218 (2)	3743 (3)	102 (2)

* Equivalent isotropic U calculated from anisotropic U.

Table 2. *Bond lengths* (Å), *bond angles and torsion angles* (°)

C(1)—C(2)	1·355 (8)*	C(1)—C(9a)	1·433 (7)
C(2)—C(3)	1·413 (8)	C(3)—C(4)	1·340 (8)
C(4)—C(4a)	1·421 (7)	C(4a)—C(9a)	1·431 (7)
C(5)—C(6)	1·357 (7)	C(6)—C(7)	1·415 (8)
C(7)—C(8)	1·349 (9)	C(8)—C(8a)	1·425 (7)
C(8a)—C(9)	1·400 (7)	C(9)—C(9a)	1·414 (6)
C(9)—C(91)	1·519 (6)	C(10)—C(4a)	1·381 (8)
C(10)—C(10a)	1·391 (7)	C(10a)—C(5)	1·424 (7)
C(10a)—C(8a)	1·438 (7)	C(91)—C(92)	1·527 (6)
C(92)—C(93)	1·521 (7)		

C(2)—C(1)—C(9a)	121·6 (5)	C(1)—C(2)—C(3)	120·7 (5)
C(2)—C(3)—C(4)	120·1 (5)	C(3)—C(4)—C(4a)	121·3 (5)
C(4)—C(4a)—C(9a)	119·8 (5)	C(4)—C(4a)—C(10)	120·3 (5)
C(9a)—C(4a)—C(10)	119·9 (5)	C(6)—C(5)—C(10a)	121·4 (5)
C(5)—C(6)—C(7)	119·4 (6)	C(6)—C(7)—C(8)	121·2 (6)
C(7)—C(8)—C(8a)	121·7 (5)	C(8)—C(8a)—C(9)	123·3 (5)
C(8)—C(8a)—C(10a)	117·1 (5)	C(9)—C(8a)—C(10a)	119·6 (5)
C(8a)—C(9)—C(9a)	119·9 (5)	C(8a)—C(9)—C(91)	120·3 (4)
C(9a)—C(9)—C(91)	119·7 (5)	C(1)—C(9a)—C(4a)	116·6 (5)
C(1)—C(9a)—C(9)	123·8 (5)	C(4a)—C(9a)—C(9)	119·6 (5)
C(4a)—C(10)—C(10a)	121·2 (5)	C(5)—C(10a)—C(8a)	119·1 (5)
C(5)—C(10a)—C(10)	121·1 (5)	C(8a)—C(10a)—C(10)	119·7 (5)
C(9)—C(91)—C(92)	113·9 (4)	C(91)—C(92)—C(93)	111·4 (4)

C(9a)—C(1)—C(2)—C(3)	−0·1 (8)	C(2)—C(1)—C(9a)—C(4a)	0·3 (7)
C(2)—C(1)—C(9a)—C(9)	−178·9 (5)	C(1)—C(2)—C(3)—C(4)	−0·3 (8)
C(2)—C(3)—C(4)—C(4a)	0·6 (8)	C(3)—C(4)—C(4a)—C(9a)	−0·3 (8)
C(3)—C(4)—C(4a)—C(10)	178·6 (5)	C(4)—C(4a)—C(9a)—C(1)	−0·1 (7)
C(4)—C(4a)—C(9a)—C(9)	179·1 (5)	C(10)—C(4a)—C(9a)—C(1)	−179·0 (5)
C(10)—C(4a)—C(9a)—C(9)	0·2 (7)	C(4)—C(4a)—C(10)—C(10a)	−177·8 (5)
C(9a)—C(4a)—C(10)—C(10a)	1·2 (7)	C(10a)—C(5)—C(6)—C(7)	−1·1 (8)
C(6)—C(5)—C(10a)—C(8a)	0·6 (7)	C(6)—C(5)—C(10a)—C(10)	−177·0 (5)
C(5)—C(6)—C(7)—C(8)	0·5 (8)	C(6)—C(7)—C(8)—C(8a)	0·5 (8)
C(7)—C(8)—C(8a)—C(9)	178·4 (5)	C(7)—C(8)—C(8a)—C(10a)	−0·9 (7)
C(8)—C(8a)—C(9)—C(9a)	−176·7 (5)	C(8)—C(8a)—C(9)—C(91)	1·2 (7)
C(10a)—C(8a)—C(9)—C(9a)	2·7 (7)	C(10a)—C(8a)—C(9)—C(91)	−179·4 (5)
C(8)—C(8a)—C(10a)—C(5)	0·4 (7)	C(8)—C(8a)—C(10a)—C(10)	178·0 (5)
C(9)—C(8a)—C(10a)—C(5)	−179·0 (5)	C(9)—C(8a)—C(10a)—C(10)	−1·4 (7)
C(8a)—C(9)—C(9a)—C(1)	177·1 (5)	C(8a)—C(9)—C(9a)—C(4a)	−2·1 (7)
C(91)—C(9)—C(9a)—C(1)	−0·8 (7)	C(91)—C(9)—C(9a)—C(4a)	−180·0 (4)
C(8a)—C(9)—C(91)—C(92)	−88·1 (6)	C(9a)—C(9)—C(91)—C(92)	89·8 (5)
C(4a)—C(10)—C(10a)—C(5)	177·0 (5)	C(4a)—C(10)—C(10a)—C(8a)	−0·6 (7)
C(9)—C(91)—C(92)—C(93)	178·7 (5)		

* These bond lengths are uncorrected. Rigid-body libration corrections: 0·006 Å for C(4)—C(4a) and C(8)—C(8a), 0·004 Å for C(91)—C(92), otherwise 0·005 Å.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44417 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

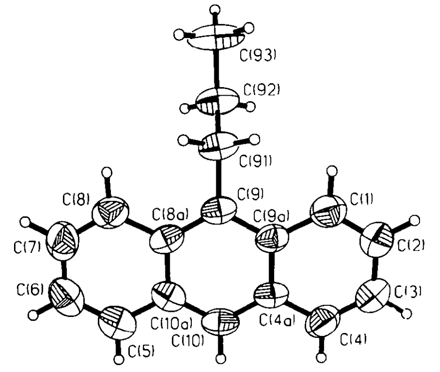

Fig. 1. Thermal-ellipsoid plot (50% level) of the title compound, showing the atom-numbering scheme. H-atom radii are arbitrary.

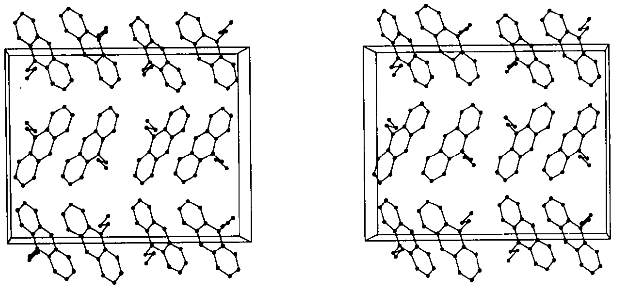

Fig. 2. Stereographic packing diagram of the title compound. View direction along *x*, *y* horizontal, *z* vertical. H atoms are omitted for clarity.

average of the bond distances C(8a)—C(9) and C(9)—C(9a) [1·407 (5) Å] is marginally larger than in anthracene [1·396 (3) Å], but substantially larger than the average of C(4a)—C(10) and C(10)—C(10a) [1·386 (6) Å]. The bond angles show this effect even more clearly. The bond angles C(1)—C(9a)—C(9) [123·8 (5)°] and C(8)—C(8a)—C(9) [123·3 (5)°] are larger than C(4)—C(4a)—C(10) [120·3 (5)°] and C(5)—C(10a)—C(10) [121·1 (5)°]. The in-plane bend of the linear anthracene skeleton [about an axis in the midpoint of C(9)—C(10) and perpendicular to the molecular plane] can also be described in terms of the diagonals of the peripheral rings, C(1)—C(4) and C(5)—C(8), which are parallel in anthracene, but make an angle of 2·4 (5)° in the title molecule.

We also observe a slight folding of the molecule along the axis C(9)—C(10). The angle between the two outer aromatic ring planes is 3·6 (5)°. A similar effect is noted in the 9-methylanthracene molecule.

The deviations of the aromatic C atoms from coplanarity, which are less than 0·01 Å in anthracene and 9,10-dimethylanthracene (Iball & Low, 1974), increase to 0·06 Å in the present case (r.m.s. 0·038 Å). This is similar to the distortion from planarity in 9-methylanthracene (0·01–0·08 Å).

The bond lengths are largely normal compared with anthracene. The exocyclic bond C(9)—C(91) is somewhat larger (2–3σ) than in 9-methylanthracene, but compares well with the corresponding value in another *n*-propyl-substituted aromatic compound that we have studied recently (Schrumpf & Jones, 1988).

The packing of 9-propylanthracene (Fig. 2) is different from that in 9-methyl-, 9,10-dimethyl- and unsubstituted anthracene. There are no unusually close intermolecular contacts in the present structure.

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Inside-Protonated 1,8-Diazabicyclo[6.4.3]pentadecane Picrate

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Abstract. $C_{13}H_{27}N_2^+.C_6H_2N_3O_7^-$, M_r = 439·56, monoclinic, $P2_1/c$, a = 11·946 (3), b = 22·900 (8), c = 7·754 (2) Å, β = 102·98 (2)°, V = 2067 (1) Å^3, Z = 4, D_x = 1·41 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·02 cm^{-1}, F(000) = 936, T = 196 K, R = 0·043 for 3084 unique observed reflections. The title compound

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