ing to an ideal tetrahedral angle. The average B—C_{ph} bond distance is 1·66 (3) Å. These bond parameters compare well with those obtained in other tetraphenylborates reported (Blundell & Powell, 1971; Cromer & Huneke, 1978; Duggan & Hendrickson, 1974; Bacci & Chilardi, 1974; Sime, Dodge, Zalkin & Templeton, 1971; Glidewell & Holden, 1982).

The B atom deviates significantly from the plane of the phenyl rings, the maximum deviation being 0·83 (2) Å. The four phenyl groups are planar to within 0·029 (2), 0·006 (2), 0·027 (3) and 0·016 (2) Å.

Intermolecular features. There are some short contacts between the cation and the anion of length less than 3·7 Å, namely C(10)···C(76) 3·60 (3), C(64)···C(36) 3·65 (4), C(65)···C(36) 3·69 (4) Å.

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The Structure of 7,14-Di-*n*-propylacenaphtho[1,2-*k*]fluoranthene

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Abstract. $C_{32}H_{26}$, $M_r = 410{\cdot}26$, monoclinic, $C2/c$, $a = 18{\cdot}727(3)$, $b = 5{\cdot}149(2)$, $c = 24{\cdot}078(4)$ Å, $\beta = 109{\cdot}67(1)^\circ$, $V = 2186{\cdot}4(4)$ Å^3, $D_m = 1{\cdot}26(1)$ (by flotation in $ZnSO_4$ solution), $D_x = 1{\cdot}247$ Mg m^{-3}, $\mu(\mathrm{Cu}\ K\alpha) = 0{\cdot}50$ mm^{-1}, $Z = 4$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $F(000) = 872$. Final $R = 0{\cdot}048$ for 1717 observed diffractometer data. The molecule is nonplanar and is closely related to 7,14-di-*n*-butylacenaphtho[1,2-*k*]fluoranthene and 7,14-di-*n*-pentylacenaphtho[1,2-*k*]fluoranthene. The propyl group is almost planar [to within ±0·035 (3) Å] forming a zigzag arrangement pointing along **c** and perpendicular to the needle axis **b**. It has normal configuration and dimensions, with C—C distances varying from 1·505 (3) to 1·531 (4) Å. The atoms of the fluoranthene moiety are coplanar to within ±0·050 (2) Å; the plane of the propyl group is almost perpendicular to it. Molecules are held together by van der Waals interactions. Bond lengths and angles are in reasonable agreement with those obtained in other 7,14-disubstituted acenaphtho[1,2-*k*]fluoranthenes.

Introduction. The crystal structure of the medium-sized nonalternant hydrocarbon fluoranthene has already been determined by X-ray and neutron diffraction (Hazell, Jones & Sowden, 1977). The structures of the 7,14-disubstituted acenaphtho[1,2-*k*]fluoranthenes (I), (II) and (III), determined from X-ray photographic and counter diffractometer data (Seth & Chakraborty,

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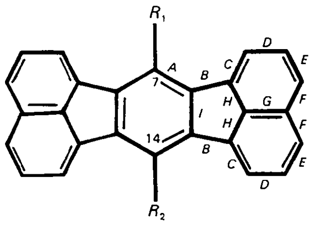
1981, 1982, 1983), have already been reported. The present work describes an X-ray structural study of compound (IV), in order to investigate the degree of planarity of (IV) compared with other related compounds.



(I) $R_1 = R_2 = n\text{-}C_4H_9$
(II) $R_1 = C_6H_5, R_2 = CH_3$
(III) $R_1 = R_2 = n\text{-}C_5H_{11}$
(IV) $R_1 = R_2 = n\text{-}C_3H_7$

Experimental. A convenient one-step synthesis of (IV) involved a Diels–Alder cycloaddition reaction between 1-chloroacenaphthylene and 7,9-di-*n*-propyl-8*H*-cyclopent[*a*]acenapthylen-8-one (Banerjee & Bhattacharya, 1977). Yellow acicular crystals, elongated along [010], by slow evaporation from acetone/benzene at room temperature, approximate lattice constants from rotation and Weissenberg photographs, accurate values by least-squares treatment of 64 randomly chosen reflections with $15 < \theta < 69°$, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, crystal *ca* 0·30 × 0·25 × 0·25 mm; intensity data up to $\theta = 70°$, index range $-22 \leq h \leq 21$, $0 \leq k \leq 6$, $0 \leq l \leq 28$, ω–2θ scans; three standard reflections monitored periodically, no significant variation; intensities were corrected for Lorentz, polarization and absorption effects (min. and max. correction factors = 0·9795, 0·9970); 2119 independent reflections, 1717 observed $[I \geq 2\sigma(I)]$.

The observed systematic absences indicated possible space groups as *Cc* or *C*2/*c*; for $Z = 4$ the molecules would be in general positions in the former, and have molecular and crystallographic symmetry coincident in the latter. The initial solution of the structure was accomplished by direct methods using *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) assuming space group *Cc*. An *E* map was generated from 228 reflections ($E \geq 1·59$) which revealed the positions of 13 C atoms. Model completed by successive weighted Fourier syntheses. Bond lengths and bond angles suggested a molecular centre of symmetry and the molecule was then shifted and refinement proceeded in space group *C*2/*c*.

Isotropic full-matrix least-squares refinement gave $R \simeq 0·13$. The positions of H atoms, fixed by trigonal geometry, were included in calculated positions (C—H = 0·95 Å) and kept fixed during refinement; positions of the remaining methyl H atoms were located from a difference Fourier synthesis. Block-diagonal least-squares refinement, including anisotropic thermal parameters for C and with all H atoms fixed. Final $R = 0·048$, $wR = 0·045$, $S = 0·998$, for 1717 observed reflections; function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = [1/\sigma(F_o)]^2$ (from counting statistics). There was a zero shift/e.s.d. in the final least-squares cycle. The final difference map was featureless, the maximum peak being 0·2 and the minimum −0·3 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on the Burroughs B6700 Computer at the Regional Computer Centre, Jadavpur University Campus, Calcutta, with program *XRAYARC* (Vickery, Bright & Mallinson, 1971). Programs *EXFFT* and *SEARCH* of *MULTAN*78 used for weighted Fourier synthesis.

Table 1. *Final atomic parameters* (× 10^4) *and equivalent isotropic temperature factors,* B_{eq}, *with e.s.d.'s in parentheses*

The deviations (Å) of the C atoms from the fluoranthene plane* are given in the last column.

$$B_{eq} = (B_{11} + B_{22} + B_{33})/3.$$

	x	y	z	B_{eq}(Å^2)	Deviations
C(1)	1312 (2)	7447 (7)	−1541 (1)	6·9	
C(2)	1123 (1)	8732 (5)	−1034 (1)	4·5	
C(3)	458 (1)	7388 (5)	−917 (1)	3·1	0·050 (2)
C(4)	227 (1)	8715 (4)	−447 (1)	2·8	0·025 (2)
C(5)	537 (1)	8019 (4)	147 (1)	2·7	−0·012 (2)
C(6)	1110 (1)	6023 (4)	441 (1)	2·9	−0·033 (2)
C(7)	1542 (1)	4232 (5)	268 (1)	3·6	−0·047 (2)
C(8)	2017 (1)	2520 (5)	694 (1)	3·9	0·001 (2)
C(9)	2076 (1)	2607 (5)	1278 (1)	3·8	0·033 (2)
C(10)	1663 (1)	4472 (5)	1477 (1)	3·2	0·003 (2)
C(11)	1665 (1)	4806 (5)	2063 (1)	4·0	0·030 (3)
C(12)	1216 (1)	6663 (5)	2177 (1)	4·4	0·030 (3)
C(13)	729 (1)	8322 (5)	1736 (1)	3·7	0·014 (3)
C(14)	721 (1)	8046 (4)	1161 (1)	2·9	−0·022 (2)
C(15)	305 (1)	9276 (4)	586 (1)	2·8	−0·021 (2)
C(16)	1192 (1)	6120 (4)	1048 (1)	2·9	−0·028 (2)

* Equation referred to orthogonal axes: $0·7278x + 0·6689y + 0·1514z - 3·4458 = 0$.

Discussion. The final atomic parameters are given in Table 1.* The labelling sequence of the asymmetric unit projected normal to **a** is shown in Fig. 1. The molecular centre of symmetry coincides with that of the unit cell. Bond lengths and angles (Table 2) in the fluoranthene ring are consistent with those in the related compounds (Hazell *et al.*, 1977; Seth & Chakraborty, 1981, 1982, 1983) and those for the picryl bromide complex (Herbstein & Kaftory, 1975). A comparison of bond lengths in compounds (I)–(IV) with values for fluoranthene, and with the results of several theoretical calculations, are given in Table 3. The 14 fluoranthene ring atoms are within ±0·050 (2) Å of their least-

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes calculations, H-atom coordinates and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44726 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·531 (4)	C(9)–C(10)	1·415 (3)
C(2)–C(3)	1·531 (3)	C(10)–C(11)	1·419 (3)
C(3)–C(4)	1·505 (3)	C(10)–C(16)	1·398 (3)
C(4)–C(5)	1·398 (3)	C(11)–C(12)	1·361 (3)
C(5)–C(6)	1·482 (3)	C(12)–C(13)	1·427 (3)
C(5)–C(15)	1·425 (3)	C(13)–C(14)	1·387 (3)
C(6)–C(7)	1·379 (3)	C(14)–C(15)	1·483 (3)
C(6)–C(16)	1·419 (3)	C(14)–C(16)	1·413 (3)
C(7)–C(8)	1·415 (3)	C(4)–C(15′)	1·396 (3)
C(8)–C(9)	1·375 (3)		

C(1)–C(2)–C(3)	111·8 (2)	C(11)–C(10)–C(16)	116·8 (2)
C(2)–C(3)–C(4)	113·0 (2)	C(10)–C(11)–C(12)	119·4 (2)
C(3)–C(4)–C(5)	121·8 (2)	C(11)–C(12)–C(13)	123·6 (2)
C(4)–C(5)–C(6)	130·6 (2)	C(12)–C(13)–C(14)	118·1 (2)
C(4)–C(5)–C(15)	121·1 (2)	C(13)–C(14)–C(15)	135·6 (2)
C(6)–C(5)–C(15)	108·3 (2)	C(13)–C(14)–C(16)	117·8 (2)
C(5)–C(6)–C(7)	136·4 (2)	C(15)–C(14)–C(16)	106·6 (2)
C(5)–C(6)–C(16)	106·0 (2)	C(5)–C(15)–C(14)	107·6 (2)
C(7)–C(6)–C(16)	117·6 (2)	C(6)–C(16)–C(10)	124·3 (2)
C(6)–C(7)–C(8)	119·2 (2)	C(6)–C(16)–C(14)	111·5 (2)
C(7)–C(8)–C(9)	122·4 (2)	C(10)–C(16)–C(14)	124·2 (2)
C(8)–C(9)–C(10)	120·3 (2)	C(5)–C(4)–C(15′)	116·9 (2)
C(9)–C(10)–C(11)	126·9 (2)	C(5)–C(15)–C(4′)	122·0 (2)
C(9)–C(10)–C(16)	116·3 (2)		

Table 3. *Comparison of mean values of bond lengths of* 7,14-*disubstituted acenaphtho*[1,2-*k*]*fluoranthenes along with the mean values of observed and theoretical values of fluoranthene* (*e.s.d.'s are the means of the individual values*).

See scheme for bond nomenclature.

					Fluoranthene	
Bond	(I)	(II)	(III)	(IV)	Mean observed	Mean theoretical*
A	1·391 (7)	1·396 (3)	1·403 (3)	1·398 (3)	1·384 (4)	1·403
B	1·472 (7)	1·480 (3)	1·482 (3)	1·483 (3)	1·476 (4)	1·461
C	1·373 (8)	1·378 (4)	1·378 (3)	1·383 (3)	1·367 (5)	1·388
D	1·424 (9)	1·419 (4)	1·421 (3)	1·421 (3)	1·411 (5)	1·414
E	1·364 (9)	1·366 (4)	1·373 (3)	1·368 (3)	1·368 (5)	1·382
F	1·411 (8)	1·418 (3)	1·421 (3)	1·417 (3)	1·422 (4)	1·424
G	1·385 (7)	1·398 (3)	1·387 (3)	1·398 (3)	1·400 (4)	1·410
H	1·415 (7)	1·414 (3)	1·416 (3)	1·416 (3)	1·411 (4)	1·428
I	1·430 (7)	1·423 (3)	1·423 (3)	1·425 (3)	1·417 (4)	1·410

* Theoretical calculations: Warren & Yandle (1968); Dewar & Trinajstic (1970); Kolc, Thulstrup & Michl (1974).

squares plane, and χ^2 values indicate non-planarity. The propyl group is planar to within ±0·035 (3) Å, forming a zigzag chain pointing along **c** and perpendicular to the needle axis (**b**). Within experimental error the propyl plane is almost perpendicular (88·2°) to the fluoranthene plane. The five-membered ring is essentially planar with maximum deviation of 0·006 (2) Å [C(5)] and is tilted slightly (0·4°) from the fluoranthene plane. Bond distances and angles of the five-membered ring show slight distortion from an ideal pentagon. The naphthalene ring is almost planar [to within ±0·016 (2) Å]; bond alternation is similar to that in naphthalene itself. The strain introduced by the presence of the five-membered ring is propagated throughout the molecule so that angle C(9)–C(10)–C(11) is significantly greater than 120° and C(5)–C(4)–C(15) is smaller than 120°. A similar result has been obtained in 6b,10b-dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene (Hazell & Hazell, 1977). This study shows that there is little distortion of the title molecule, which is almost symmetrical about the C(10)–C(16) vector as in fluoranthene itself. Bond lengths and angles suggest that all bonds in the ring nucleus are involved in resonance.

The discrepancies of the bond lengths (*B,C,H,I*) in (I)–(IV) (Table 3) are due to the presence of the fused

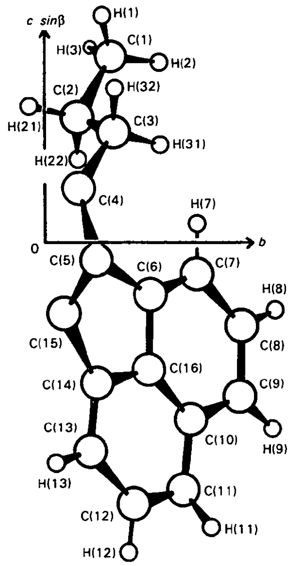

Fig. 1. View of the asymmetric unit along **a** and the atom-numbering scheme.

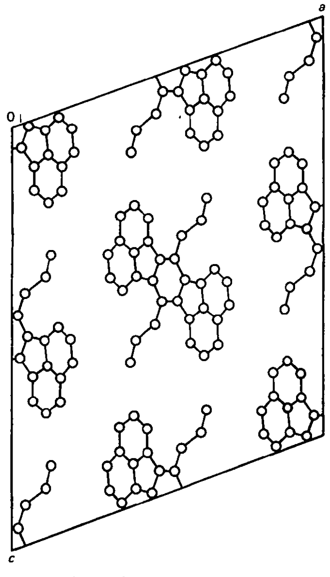

Fig. 2. Molecular packing viewed down **b**. H atoms have been omitted for clarity.

five-membered ring, and also to the planar configuration of the molecule, which produces some unusual bond lengths and angles. The planarity of the fluoranthene moiety is found to be similar for compounds (I), (III) and (IV), whilst it is significantly non-planar in (II).

Fig. 2 shows the content of the unit cell projected down **b**. The molecules are packed in layers, but are displaced with respect to one another to relieve short intermolecular contacts, so that packing forces are unlikely to influence the geometry of the molecule to any appreciable extent.

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Endoannular Reactions and Ring–Chain Tautomerism. Structure of 1-(2-Hydroxyethyl)-3-methyl-2,4(1*H*,3*H*)-quinazolinedione

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Abstract. $C_{11}H_{12}N_2O_3$, $M_r = 220{\cdot}2$, tetragonal, $P\bar{4}2_1c$, $a = 17{\cdot}008\ (5)$, $c = 7{\cdot}081\ (3)$ Å, $V = 2048\ (1)$ Å^3, $Z = 8$, $D_x = 1{\cdot}43$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 0{\cdot}9$ mm^{-1}, $F(000) = 928$, room temperature, $R = 0{\cdot}044$ for 1023 reflections. The reaction of 2-(2-bromoethyl)isatoic anhydride with methylamine affords the title compound, which is the ring–chain tautomer of 2,3-dihydro-6-methyl-4,1,6-benzoxadiazonine-5,7(1*H*,6*H*)-dione. The side-chain hydroxyl group is disordered in the crystal assuming two conformations, approximately *trans* and *gauche**, both involved in the hydrogen-bonding network of the crystal packing.

Introduction. In order to investigate the endoannular interactions and ring–chain tautomerism in medium-sized heterocycles (Pinnen, Zanotti & Lucente, 1982; Zanotti, Pinnen, Lucente, Cerrini, Gavuzzo & Mazza, 1983; Pinnen, Zanotti, Lucente, Cerrini, Fedeli & Gavuzzo, 1985) we tried to synthesize the nine-membered system 2,3-dihydro-6-methyl-4,1,6-benzoxadiazonine-5,7(1*H*,6*H*)-dione (I) by reacting 2-(2-bromoethyl)isatoic anhydride with methylamine according to the procedure of Barcza, Coppola & Shapiro (1979). The nine-membered ring of compound (I) contains in fact a free amino group and a potentially reactive *N*-acylurethane carbonyl suitably located to study the endoannular reaction and related tetrahedral intermediates. As previously reported (Pinnen, Lucente, Mazza & Pochetti, 1987) this reaction leads to

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