Acta Cryst. (1983). C**39**, 625–627

The Structure of 7,14-Di-*n*-pentylacenaphtho[1,2-*k*]fluoranthene, $C_{36}H_{34}$

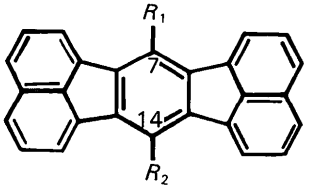
By S. Seth and S. Chakraborty

Department of Physics, University of Burdwan, Burdwan-713 104, *WB, India*

(*Received* 7 *September* 1982; *accepted* 5 *January* 1983)

Abstract. $M_r = 466{\cdot}7$, monoclinic $P2_1/c$, $a = 5{\cdot}071$ (3), $b = 17{\cdot}971$ (4), $c = 14{\cdot}156$ (4) Å, $\beta = 90{\cdot}20$ (4)°, $V = 1290{\cdot}02$ Å^3, $D_m = 1{\cdot}21$ (1) (by flotation in $ZnSO_4$ solution), $D_x = 1{\cdot}201$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·074 mm^{-1}, $Z = 2$, λ(Mo $K\alpha$) = 0·71069 Å, $F(000) = 500$, $T = 298$ K. Final $R = 0{\cdot}048$ for 1842 observed diffractometer data. The structure is closely related to that of 7,14-dibutylacenaphtho[1,2-*k*]-fluoranthene. The atoms of the fluoranthene ring are coplanar to within ±0·034 (2) Å. The pentyl group, forming a zigzag arrangement parallel to the needle axis (*a*), is almost planar and has normal configuration and dimensions, with C—C distances 1·505 (3)–1·535 (3) Å. It is perpendicular to the fluoranthene moiety. The dipentylacenaphthofluoranthene molecules are held together by van der Waals forces. The molecular parameters are in reasonably good agreement with those found in similar structures.

Introduction. The crystal structure of fluoranthene, a medium-sized nonalternant hydrocarbon, has been determined by X-ray and neutron diffraction (Hazell, Jones & Sowden, 1977) and that of (I) and (II) from X-ray photographic and counter diffractometer data (Seth & Chakraborty, 1981, 1982). The present investigation of the title compound (III) by X-ray methods is the extension of this series to $R_1 = R_2 =$ pentyl in order to investigate the effect of substitution of aliphatic chains on the planarity of the fluoranthene moiety, and also to study the bonding in the molecule.



(I) $R_1 = R_2 = C_4H_9$
(II) $R_1 = C_6H_5$, $R_2 = CH_3$
(III) $R_1 = R_2 = C_5H_{11}$

Experimental. The title compound was conveniently synthesized by Banerjee & Bhattacharya (1977) by a Diels–Alder cycloaddition reaction between 1-chloroacenaphthylene and 7,9-di-*n*-pentyl-8*H*-cyclopent[*a*]-acenaphthylen-8-one. Needle-shaped, deep-yellow crystals by slow evaporation from acetone/benzene at room temperature; approximate lattice constants from rotation and Weissenberg photographs, accurate values by least squares from the 2θ values of 25 reflexions, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, crystal *ca* 0·45 × 0·42 × 0·40 mm; intensity data for $4{\cdot}6 \le 2\theta \le 50°$, ω–2θ scans; three standard reflexions monitored periodically did not vary significantly during data collection; Lorentz–polarization corrections but not absorption; 2256 independent reflexions, 1844 observed [$I \ge 2\sigma(I)$]; structure solved direct methods [*MULTAN* (Germain, Main & Woolfson, 1971)] with 172 $|E|$ values <1·8 for phase determination; following isotropic full-matrix least-squares refinement, the H atoms (excluding methyl H atoms) were included in calculated positions (C—H = 0·95 Å); the positions of the methyl H atoms were then located from a difference Fourier synthesis; each H atom was given the isotropic temperature factor of its associated C atom; block-diagonal least-squares refinement, including anisotropic thermal parameters for C and varying only the positional parameters of the H atoms, was discontinued when all the parameter shifts were less than the corresponding e.s.d.'s; final R and R_w omitting two reflexions because of obvious strong secondary extinctions = 0·048 and 0·038 for 1842 reflexions; function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = [1/\sigma(F_o)]^2$ (from counting statistics); final difference map was featureless, the maximum being 0·1 e Å^{-3} and the minimum −0·2 e Å^{-3}; scattering factors from *International Tables for X-ray Crystallography* (1974); all computations were performed on the Burroughs B 6700 computer of the Regional Computer Centre, Jadavpur University Campus, Calcutta with the program *XRAY ARC* (Vickery, Bright & Mallinson, 1971).

Discussion. The final atomic coordinates are given in Table 1.* The asymmetric unit of the title molecule (III) 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

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms and details of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38327 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/83/050625-03$01.50 © 1983 International Union of Crystallography

Table 1. *Positional parameters* ($\times 10^4$ *for* C, $\times 10^3$ *for* H) *with e.s.d.'s in parentheses, and temperature factors,* B_{eq} *for* C, B_{iso} *for* H

The deviations (Å) of the C atoms from the fluoranthene plane* are given in the last columns. $B_{eq} = (B_{11} + B_{22} + B_{33})/3$.

	x	y	z	B_{eq} or B (Å^2)	Deviations
C(1)	12585 (5)	−1024 (1)	5196 (2)	6·7	
C(2)	11252 (5)	−981 (1)	4234 (1)	5·3	
C(3)	12550 (4)	−442 (1)	3548 (1)	4·4	
C(4)	11171 (4)	−426 (1)	2591 (1)	3·9	
C(5)	12534 (4)	88 (1)	1876 (1)	3·8	0·024 (2)
C(6)	11247 (4)	46 (1)	918 (1)	3·1	−0·008 (2)
C(7)	9233 (4)	538 (1)	647 (1)	3·1	−0·003 (2)
C(8)	7957 (4)	1153 (1)	1173 (1)	3·3	0·014 (2)
C(9)	8165 (4)	1449 (1)	2072 (1)	4·1	−0·005 (2)
C(10)	6480 (4)	2047 (1)	2326 (2)	4·9	−0·017 (2)
C(11)	4650 (4)	2345 (1)	1717 (2)	4·9	−0·011 (2)
C(12)	4395 (4)	2047 (1)	790 (1)	4·0	0·003 (2)
C(13)	2593 (4)	2286 (1)	85 (2)	4·7	−0·003 (2)
C(14)	2574 (4)	1940 (1)	−780 (2)	4·6	0·007 (2)
C(15)	4299 (4)	1342 (1)	−1004 (1)	3·8	0·019 (2)
C(16)	6025 (4)	1090 (1)	−327 (1)	3·2	−0·004 (2)
C(17)	8028 (4)	493 (1)	−261 (1)	3·1	−0·034 (2)
C(18)	6061 (4)	1464 (1)	555 (1)	3·3	0·017 (2)
H(1)	1253 (4)	−51 (1)	549 (1)	6·0	
H(2)	1160 (4)	−136 (1)	563 (1)	6·0	
H(3)	1440 (4)	−118 (1)	515 (1)	6·0	
H(21)	940 (4)	−84 (1)	432 (1)	5·4	
H(22)	1117 (4)	−148 (1)	392 (1)	5·4	
H(31)	1446 (4)	−60 (1)	345 (1)	4·4	
H(32)	1260 (4)	7 (1)	384 (1)	4·4	
H(41)	927 (4)	−27 (1)	267 (1)	4·0	
H(42)	1109 (4)	−94 (1)	231 (1)	4·0	
H(51)	1440 (4)	−5 (1)	181 (1)	3·6	
H(52)	1250 (3)	60 (1)	212 (1)	3·6	
H(9)	946 (4)	124 (1)	254 (1)	4·3	
H(10)	673 (4)	227 (1)	297 (1)	4·9	
H(11)	344 (4)	276 (1)	192 (1)	5·0	
H(13)	133 (4)	269 (1)	22 (1)	5·0	
H(14)	129 (4)	211 (1)	−128 (1)	4·7	
H(15)	421 (4)	111 (1)	−164 (1)	4·0	

* See deposition footnote.

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

C(1)–C(2)	1·520 (3)	C(10)–C(11)	1·373 (3)
C(2)–C(3)	1·522 (3)	C(11)–C(12)	1·423 (3)
C(3)–C(4)	1·523 (3)	C(12)–C(13)	1·418 (3)
C(4)–C(5)	1·535 (3)	C(12)–C(18)	1·387 (3)
C(5)–C(6)	1·505 (3)	C(13)–C(14)	1·373 (3)
C(6)–C(7)	1·403 (3)	C(14)–C(15)	1·422 (3)
C(7)–C(8)	1·483 (3)	C(15)–C(16)	1·372 (3)
C(7)–C(17)	1·423 (3)	C(16)–C(17)	1·480 (3)
C(8)–C(9)	1·383 (3)	C(16)–C(18)	1·419 (3)
C(8)–C(18)	1·413 (3)	C(6)–C(17′)	1·394 (3)
C(9)–C(10)	1·420 (3)		
C(1)–C(2)–C(3)	114·4 (2)	C(11)–C(12)–C(18)	116·9 (2)
C(2)–C(3)–C(4)	112·4 (2)	C(13)–C(12)–C(18)	116·8 (2)
C(3)–C(4)–C(5)	113·0 (2)	C(12)–C(13)–C(14)	119·6 (2)
C(4)–C(5)–C(6)	111·7 (2)	C(13)–C(14)–C(15)	122·6 (2)
C(5)–C(6)–C(7)	121·9 (2)	C(14)–C(15)–C(16)	119·1 (2)
C(6)–C(7)–C(8)	130·6 (2)	C(15)–C(16)–C(17)	136·1 (2)
C(6)–C(7)–C(17)	121·4 (2)	C(15)–C(16)–C(18)	117·6 (2)
C(8)–C(7)–C(17)	108·0 (2)	C(17)–C(16)–C(18)	106·3 (2)
C(7)–C(8)–C(9)	135·8 (2)	C(7)–C(17)–C(16)	108·0 (2)
C(7)–C(8)–C(18)	106·3 (2)	C(8)–C(18)–C(12)	124·3 (2)
C(9)–C(8)–C(18)	117·9 (2)	C(8)–C(18)–C(16)	111·4 (2)
C(8)–C(9)–C(10)	118·7 (2)	C(12)–C(18)–C(16)	124·3 (2)
C(9)–C(10)–C(11)	122·9 (2)	C(7)–C(6)–C(17′)	116·7 (2)
C(10)–C(11)–C(12)	119·4 (2)	C(7)–C(17)–C(6′)	121·9 (2)
C(11)–C(12)–C(13)	126·3 (2)		

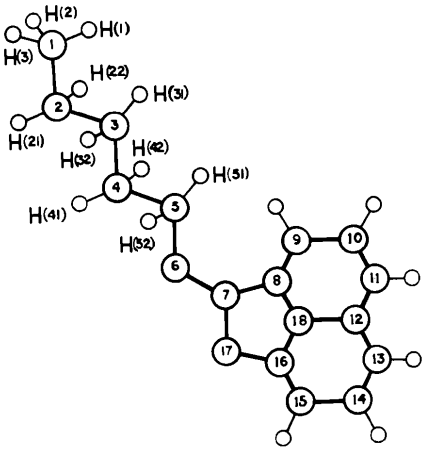


Fig. 1. Asymmetric unit of the title molecule showing the numbering scheme. The H atoms in fluoranthene, represented by small circles, have the same number as the C atoms to which they are attached.

with those in related compounds (Hazell *et al.*, 1977; Seth & Chakraborty, 1981, 1982). The atoms of the fluoranthene ring are within ±0·034 (2) Å of their least-squares plane. The deviation from planarity for (III) is larger than that for (I) [±0·025 (6) Å] while the ring deviates significantly for (II) [±0·101 (3) Å] which may be due to the difference in environments. The χ^2 value [$\sum_{m=1}^{\Omega} d_m^2/\sigma_{ref}^2$ where d_m is the deviation from the least-squares plane and σ_{ref}^2 is the mean-square value of $\sigma_{x,y,z}$] for the fluoranthene ring is 874, *i.e.* the fluoranthene ring is not strictly planar. The pentyl group is almost planar with maximum deviation of −0·029 (2) Å [C(3)]. It forms a zigzag arrangement elongated along **c** and parallel to the shortest axis; within experimental error it is normal (89·3°) to the plane through the fluoranthene atoms. The five-membered ring is essentially planar [to within ±0·008 (2) Å] and is tilted slightly from the fluoranthene plane. The interior angles of the five-membered ring show that it is slightly distorted from ideal pentagonal shape and there is bond alternation in the naphthalene nucleus similar to the situation in naphthalene itself.

The angle C(7)–C(6)–C(17′) is smaller than 120° and C(11)–C(12)–C(13) is significantly larger than 120°, *cf* 6b,10b-dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene (Hazell & Hazell, 1977). Some strain is therefore introduced in the fluoranthene ring due to the presence of a five-membered ring between the benzene and naphthalene nuclei and is propagated throughout the molecule. There is, therefore, distortion of the dipentylacenaphthofluoranthene molecule.

The title molecule is almost symmetrical about the line through the C(12)–C(18) bond and is non-planar. Bond lengths and angles suggest that all bonds in the molecule, except the pentyl group, are involved in resonance.

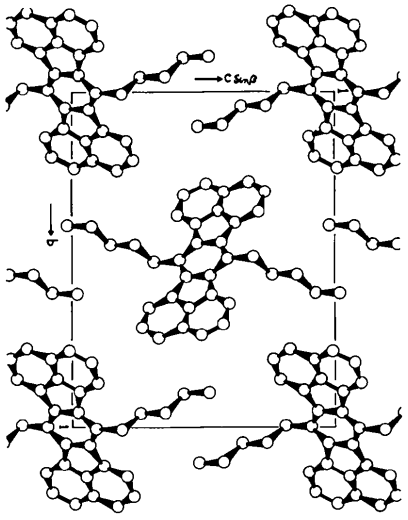


Fig. 2. Projection of the molecule in a plane normal to **a**. H atoms are omitted.

A packing diagram is shown in Fig. 2. Layers of molecules are displaced with respect to one another to relieve short intermolecular contacts. The structure consists of individual molecules packed in the crystal *via* van der Waals forces. There are no unusually short intermolecular contacts, so that packing forces are unlikely to influence the geometry of the molecule to an appreciable extent.

The authors thank Dr Siddhartha Ray, IACS, Jadavpur, Calcutta for advice on Computation and some stimulating discussions and Dr A. J. Bhattacharya of the Department of Chemistry, University of Burdwan, for supplying the sample used in this investigation. Financial assistance from the University of Burdwan is gratefully acknowledged.

References

BANERJEE, P. K. & BHATTACHARYA, A. J. (1977). *Indian J. Chem.* **15B**, 953–955.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.
HAZELL, A. C. & HAZELL, R. G. (1977). *Acta Cryst.* B**33**, 360–365.
HAZELL, A. C., JONES, D. W. & SOWDEN, J. M. (1977). *Acta Cryst.* B**33**, 1516–1522.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–73. Birmingham: Kynoch Press.
SETH, S. & CHAKRABORTY, S. (1981). *Acta Cryst.* B**37**, 1144–1146.
SETH, S. & CHAKRABORTY, S. (1982). *Acta Cryst.* B**38**, 1380–1383.
VICKERY, B. L., BRIGHT, D. & MALLINSON, P. R. (1971). *XRAY ARC. IBM* 1130 Program System for Crystallography modified for a Burroughs Computer.

Acta Cryst. (1983). C**39**, 627–630

Nucleic Acid Binding Drugs. VIII. Structures of 1-[2-(Diethylamino)ethylamino]anthracene-9,10-dione, $C_{20}H_{22}N_2O_2$ (I), and 1,5-Bis[2-(diethylamino)ethylamino]anthracene-9,10-dione, $C_{26}H_{36}N_4O_2$ (II), Models for Antitumour Drugs

BY P. ALMOND, S. D. CUTBUSH, S. A. ISLAM, R. KURODA* AND S. NEIDLE

Cancer Research Campaign Biomolecular Structure Research Group, Department of Biophysics, King's College, 26–29 Drury Lane, London WC2B 5RL, England

B. M. GANDECHA

School of Pharmacy, Leicester Polytechnic, PO Box 143, *Leicester LE*1 9*BH, England*

AND J. R. BROWN

*Department of Pharmaceutical Chemistry, Sunderland Polytechnic, Green Terrace, Sunderland SR*1 3*SD, England*

(*Received* 21 *October* 1982; *accepted* 19 *January* 1983)

Abstract. (I) $M_r = 322{\cdot}41$, $P2_1/n$, $a = 7{\cdot}118$ (1), $b = 26{\cdot}873$ (2), $c = 8{\cdot}886$ (1) Å, $\beta = 97{\cdot}74$ (1)°, $V = 1684{\cdot}3$ (6) Å^3, $Z = 4$, $D_m = 1{\cdot}27$, $D_x = 1{\cdot}271$ Mg m^{-3}, λ (Cu $K\alpha$) = 1·54178 Å, $\mu = 6{\cdot}67$ cm^{-1}, $F(000) = 688$, $T = 298$ K, $R_w = 0{\cdot}049$ for 981 unique significant reflections. (II) $M_r = 436{\cdot}61$, $P2_1/c$, $a = 15{\cdot}360$ (2), $b = 5{\cdot}245$ (1), $c = 15{\cdot}483$ (1) Å, $\beta = 94{\cdot}23$ (1)°, $V = 1244{\cdot}0$ (5) Å^3, $Z = 2$, $D_m = 1{\cdot}17$, $D_x = 1{\cdot}165$ Mg m^{-3},

* To whom correspondence should be addressed.

0108-2701/83/050627-04$01.50 © 1983 International Union of Crystallography