$$C_{17}H_{26}O_2$$

Fig. 2. Perspective view of the molecule (down **a**), showing the numbering of the nonhydrogen atoms.

involved in any hydrogen bonding show a similar bond length (Craven, Cusatis, Gartland & Vizzini, 1973). The $C(sp^3)-C(sp^3)$ bond lengths vary from 1·511 (6) to 1·547 (5) Å with an average value of 1·530 Å. In ring *D*, bonds involving O(3) show different lengths [1·439 (4), 1·413 (5) Å]. Such disproportionation has been found in other five- or six-membered rings containing oxygen (Hanson, 1977; Fujiwara & van der Veen, 1979) and in sugar molecules (Jeffrey, Pople, Binkley & Vishveshwara, 1978; Takagi, Nordenson & Jeffrey, 1979). Further, the angle at oxygen is found to be the largest within the ring (Takusagawa & Jacobson, 1978; Fujiwara & van der Veen, 1979; Takagi,

Nordenson & Jeffrey, 1979), with the angles at the two adjacent C atoms significantly larger than those at the other two C atoms (Singh & Ahmed, 1969; De Graaff, Admiraal, Koen & Romers, 1977).

One of us (PC) thanks the Indian Institute of Science for financial assistance.

References

Altona, C., Geise, H. J. & Romers, C. (1968). *Tetrahedron*, **24**, 13–32.

Bucourt, R. (1974). *Top. Stereochem.* **8**, 159–224.

Craven, B. M., Cusatis, C., Gartland, G. L. & Vizzini, E. A. (1973). *J. Mol. Struct.* **16**, 331–342.

De Graaff, R. A. G., Admiraal, G., Koen, E. H. & Romers, C. (1977). *Acta Cryst.* B**33**, 2459–2464.

Fujiwara, H. & van der Veen, J. M. (1979). *Acta Cryst.* B**35**, 1287–1290.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Hanson, A. W. (1977). *Acta Cryst.* B**33**, 889–891.

Jeffrey, G. A., Pople, J. A., Binkley, J. S. & Vishveshwara, S. (1978). *J. Am. Chem. Soc.* **100**, 373–379.

Kato, C., Konaka, S., Iijima, T. & Kimura, M. (1969). *Bull. Chem. Soc. Jpn*, **42**, 2148–2158.

Maji, S. K., Mukherjee, S. K., Mukherjee, D. & Dutta, P. C. (1981). *J. Chem. Soc. Perkin Trans.* 1. In the press.

Singh, P. & Ahmed, F. R. (1969). *Acta Cryst.* B**25**, 2401–2408.

Takagi, S., Nordenson, S. & Jeffrey, G. A. (1979). *Acta Cryst.* B**35**, 991–993.

Takusagawa, F. & Jacobson, R. A. (1978). *Acta Cryst.* B**34**, 213–218.

Structure of 7,14-Dibutylacenaphtho[1,2-*k*]fluoranthene

By S. Seth and S. Chakraborty

Department of Physics, University of Burdwan, Burdwan 713104, WB, India

Abstract. $C_{34}H_{30}$, $M_r = 438$, monoclinic, $P2_1/c$, $a = 4·704$ (2), $b = 12·52$ (1), $c = 20·11$ (1) Å, $\beta = 96·01$ (2)°, $V = 1177·87$ Å³, $Z = 2$, $D_m = 1·22$ (1), $D_c = 1·235$ Mg m⁻³, $\mu(\text{Cu } K\alpha) = 0·526$ mm⁻¹, $F(000) = 468$. The structure was solved by direct methods; final $R = 0·085$ for 1100 photographic data. The fluoranthene molecule is planar and the butyl group is parallel to the *a* axis forming a zigzag chain. The butyl group has the normal configuration and dimensions, with C—C distances varying from 1·504 (8) to 1·527 (7) Å and is nearly perpendicular to the fluor-anthene ring. Bond lengths and angles are close to normal values.

Introduction. The crystal structures of many aromatic hydrocarbons have already been investigated by X-ray analysis. The structure of the medium-sized non-alternant hydrocarbon fluoranthene has been determined by Hazell, Jones & Sowden (1977). We have determined the crystal structure of 7,14-dibutyl-acenaphtho[1,2-*k*]fluoranthene by X-ray diffraction to investigate whether or not the structure is planar and

how the butyl group is coordinated to the fluoranthene group. It was also the intention to see whether or not the fluoranthene group is composed of resonance rings.

Here we focus on the crystal structure of $C_{34}H_{30}$ which crystallizes, as brown prisms elongated along the [100] direction, on slow evaporation of a solution in benzene at room temperature. D_m was measured by flotation in aqueous $ZnSO_4$ solution. Rotation, oscillation and Weissenberg photographs of a crystal measuring $0.35 \times 0.2 \times 0.15$ mm showed monoclinic symmetry. Systematically absent reflexions ($0k0$ absent for k odd, $h0l$ absent for l odd) indicated the space group $P2_1/c$; there are two formula units per unit cell. Equi-inclination Weissenberg photographs (Cu radiation, $\lambda = 1.5418$ Å) of levels $0–3kl$ and $h0–6l$ were taken to record 1100 independent reflexions.

Integrated intensities of reflexions were estimated by eye by comparing them with a calibrated scale, and were subjected to the usual corrections for Lorentz and polarization factors, and also for spot shape, but no attempt was made to correct for absorption. Overall scale and temperature factors were obtained from a Wilson plot and the centrosymmetric space group with a hypercentric intensity distribution was confirmed by the $N(Z)$ test (Howells, Philips & Rogers, 1950).

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). Normalized structure factors, E, were calculated from a Wilson plot and 168 |E|'s greater than 1.4 were used in the phase-determination procedure. Two of the eight sets had identical values for all the figures of merit, the combined figure of merit having a value of 2.997, much higher than that of any of the remaining sets. An E map with one of these two sets was computed and all the 17 C atoms were revealed as the highest 17 peaks.

Isotropic refinement of C atoms alone gave an initial R value of 0.16. As the molecule contains a large number of H atoms, which represent a significant proportion of the total scattering power, their effect on the refinement was then considered. The positions of the H atoms, which were fixed by trigonal geometry, were calculated on the basis of a C—H distance of 1.08 Å. When the 12 H atoms with fixed positions were included, but not refined, the isotropic refinement of the C atoms alone gave an R of 0.135.

The block-diagonal approximation was used with a weighting factor of the type $w = 1/\sigma^2(F)$ with $\sigma(F)$, the standard deviation of |F_o|, assumed as follows: for |F_o| < 8.1, $\sigma(F) = (0.175|F_o| + 2.2)^{1/2}$; for |$F_o$| > 8.1, $\sigma(F) = (0.4285|F_o|)^{1/2}$. With the inclusion of the remaining three methyl H atoms, whose positions were determined from a difference Fourier map, the R value changed very slowly. All H atoms were given the same isotropic temperature factor as their associated C atoms. Anisotropic refinement of the C atoms was started with the above weighting scheme. The H atom

positions were recalculated after every two cycles and throughout the refinement layer scaling was carried out. The structure was then further refined with the C atoms treated anisotropically and varying only the positional parameters of H atoms. The final refinement resulted in a residual ($R = \sum ||F_o| - |F_c||/\sum |F_o|$) of 0.085 for observed reflexions only. The final atomic parameters are listed in Table 1.*

Structure factors were calculated with scattering factors taken from *International Tables for X-ray Crystallography* (1974).

Table 1. *Final atomic parameters*

Positional parameters are $\times 10^4$ for C atoms and $\times 10^3$ for H atoms. E.s.d.'s are in parentheses. The temperature factors, B_{eq} (equivalent) or B, and the deviations (Å) of the C atoms from the fluoranthene plane* are given in the last two columns. $[B_{eq} = (B_{11} + B_{22} + B_{33})/3.]$

	x	y	z	B_{eq} or B (Å^2)	Deviations
C(1)	−2224 (18)	4633 (5)	10988 (4)	6.6	
C(2)	−3902 (13)	3702 (5)	10658 (3)	5.0	
C(3)	−2059 (10)	2740 (4)	10572 (3)	3.8	
C(4)	−3773 (10)	1824 (4)	10227 (3)	3.5	0.015 (5)
C(5)	−1858 (10)	890 (4)	10115 (3)	3.1	−0.005 (5)
C(6)	−420 (10)	822 (4)	9547 (3)	3.2	−0.002 (5)
C(7)	−377 (11)	1566 (4)	8976 (3)	3.4	0.004 (5)
C(8)	−1573 (12)	2541 (5)	8811 (3)	4.6	−0.025 (6)
C(9)	−964 (14)	3002 (5)	8193 (3)	5.3	−0.007 (7)
C(10)	755 (14)	2544 (6)	7772 (3)	5.2	0.010 (6)
C(11)	2053 (12)	1547 (5)	7941 (3)	4.3	0.013 (6)
C(12)	3940 (13)	991 (6)	7567 (3)	5.1	−0.008 (6)
C(13)	5042 (14)	31 (6)	7795 (3)	5.4	−0.006 (7)
C(14)	4356 (12)	−427 (5)	8405 (3)	4.3	0.007 (6)
C(15)	2555 (10)	111 (4)	8786 (3)	3.5	0.005 (5)
C(16)	1393 (10)	−59 (4)	9425 (3)	3.2	−0.011 (5)
C(17)	1442 (11)	1099 (5)	8539 (3)	3.7	0.005 (5)
H(1)	−117 (15)	433 (6)	1136 (4)	5.8	
H(2)	−379 (16)	527 (6)	1115 (4)	5.8	
H(3)	−69 (15)	494 (6)	1066 (3)	5.8	
H(21)	−506 (14)	383 (5)	1024 (3)	5.2	
H(22)	−571 (14)	343 (6)	1096 (3)	5.2	
H(31)	−99 (12)	248 (5)	1103 (3)	4.0	
H(32)	−20 (13)	288 (5)	1028 (3)	4.0	
H(41)	−479 (12)	210 (5)	980 (3)	3.8	
H(42)	−540 (12)	164 (5)	1048 (3)	3.8	
H(8)	−292 (14)	289 (5)	916 (3)	4.8	
H(9)	−204 (14)	372 (6)	807 (4)	5.6	
H(10)	127 (15)	287 (6)	730 (4)	5.6	
H(12)	449 (14)	134 (6)	713 (3)	5.3	
H(13)	648 (15)	−37 (6)	754 (4)	5.5	
H(14)	523 (13)	−121 (5)	857 (3)	4.5	

* Equation referred to orthogonal axes: $-0.7369X - 0.4730Y - 0.4830Z + 8.0810 = 0.0$.

Discussion. The labelling sequence in the molecule is given in Fig. 1. The bond lengths and angles (Tables 2 and 3) in the fluoranthene rings are in good agreement with those in fluoranthene itself (Hazell, Jones & Sowden, 1977).

To study the planarity of the molecule, various mean planes were calculated. The plane through the 14 fluoranthene ring atoms shows that several atoms deviate slightly from coplanarity. The five-membered ring is tilted slightly from the mean plane. The butyl group forms a zigzag chain, elongated along the *b* axis and parallel to the needle axis (*a* axis). The fluoranthene ring is almost planar and the plane of the butyl group is almost perpendicular (87°) to it.

Due to the presence of a five-membered ring between the benzene and naphthalene nuclei, some strain is introduced which is propagated throughout the mole-

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

C(1)–C(2)	1·521 (10)	C(9)–C(10)	1·358 (9)
C(2)–C(3)	1·504 (8)	C(10)–C(11)	1·415 (9)
C(3)–C(4)	1·527 (7)	C(11)–C(12)	1·407 (8)
C(4)–C(5)	1·507 (7)	C(11)–C(17)	1·385 (8)
C(5)–C(6)	1·391 (7)	C(12)–C(13)	1·369 (10)
C(6)–C(7)	1·480 (7)	C(13)–C(14)	1·423 (9)
C(6)–C(16)	1·430 (7)	C(14)–C(15)	1·376 (8)
C(7)–C(8)	1·370 (8)	C(15)–C(16)	1·463 (7)
C(7)–C(17)	1·415 (7)	C(15)–C(17)	1·414 (8)
C(8)–C(9)	1·425 (9)	C(5)–C(16')	1·395 (7)
C(1)–H(1)	0·94 (7)	C(4)–H(42)	0·99 (6)
C(1)–H(2)	1·16 (7)	C(8)–H(8)	1·08 (6)
C(1)–H(3)	1·10 (7)	C(9)–H(9)	1·05 (7)
C(2)–H(21)	0·96 (7)	C(10)–H(10)	1·08 (7)
C(2)–H(22)	1·15 (7)	C(12)–H(12)	1·03 (7)
C(3)–H(31)	1·06 (6)	C(13)–H(13)	1·03 (7)
C(3)–H(32)	1·12 (6)	C(14)–H(14)	1·10 (7)
C(4)–H(41)	1·01 (6)		
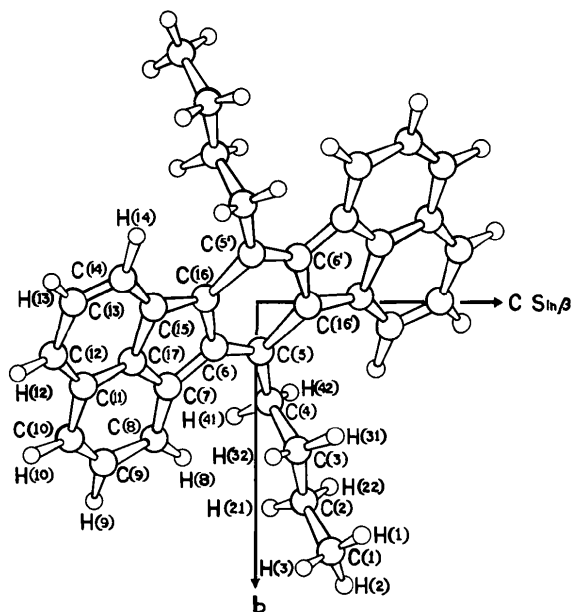
Fig. 1. The title compound projected normal to the *a* axis.

Table 3. *Bond angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)–C(3)	112·6 (5)	C(10)–C(11)–C(17)	116·2 (5)
C(2)–C(3)–C(4)	111·7 (4)	C(12)–C(11)–C(17)	117·7 (5)
C(3)–C(4)–C(5)	110·8 (4)	C(11)–C(12)–C(13)	119·6 (6)
C(4)–C(5)–C(6)	121·3 (4)	C(12)–C(13)–C(14)	122·1 (6)
C(5)–C(6)–C(7)	130·2 (5)	C(13)–C(14)–C(15)	119·5 (5)
C(5)–C(6)–C(16)	122·6 (5)	C(14)–C(15)–C(16)	136·6 (5)
C(7)–C(6)–C(16)	107·2 (4)	C(14)–C(15)–C(17)	117·3 (5)
C(6)–C(7)–C(8)	135·3 (5)	C(16)–C(15)–C(17)	106·1 (4)
C(6)–C(7)–C(17)	106·2 (4)	C(6)–C(16)–C(15)	108·8 (4)
C(8)–C(7)–C(17)	118·5 (5)	C(7)–C(17)–C(11)	124·5 (5)
C(7)–C(8)–C(9)	117·2 (5)	C(7)–C(17)–C(15)	111·7 (5)
C(8)–C(9)–C(10)	124·1 (6)	C(11)–C(17)–C(15)	123·8 (5)
C(9)–C(10)–C(11)	119·5 (6)	C(6)–C(5)–C(16')	116·3 (6)
C(10)–C(11)–C(12)	126·1 (6)	C(6)–C(16)–C(5')	121·1 (6)
C(2)–C(1)–H(1)	104 (4)	C(5)–C(4)–H(42)	115 (3)
C(2)–C(1)–H(2)	109 (4)	H(41)–C(4)–H(42)	101 (5)
C(2)–C(1)–H(3)	110 (4)	C(7)–C(8)–H(8)	117 (3)
H(1)–C(1)–H(2)	110 (6)	C(9)–C(8)–H(8)	126 (3)
H(1)–C(1)–H(3)	108 (6)	C(8)–C(9)–H(9)	114 (4)
H(2)–C(1)–H(3)	114 (5)	C(10)–C(9)–H(9)	121 (4)
C(1)–C(2)–H(21)	118 (4)	C(9)–C(10)–H(10)	126 (4)
C(1)–C(2)–H(22)	112 (3)	C(11)–C(10)–H(10)	115 (4)
C(3)–C(2)–H(21)	108 (4)	C(11)–C(12)–H(12)	118 (4)
C(3)–C(2)–H(22)	107 (3)	C(13)–C(12)–H(12)	122 (4)
H(21)–C(2)–H(22)	97 (5)	C(12)–C(13)–H(13)	120 (4)
C(2)–C(3)–H(31)	112 (3)	C(14)–C(13)–H(13)	117 (4)
C(2)–C(3)–H(32)	115 (3)	C(13)–C(14)–H(14)	121 (3)
C(4)–C(3)–H(31)	110 (3)	C(15)–C(14)–H(14)	120 (3)
C(4)–C(3)–H(32)	106 (3)	C(5)–C(4)–H(41)	112 (3)
H(31)–C(3)–H(32)	101 (5)	C(3)–C(4)–H(42)	109 (3)
C(3)–C(4)–H(41)	108 (3)		

cule so that the angle C(6)–C(5)–C(16') is smaller than 120° and C(10)–C(11)–C(12) is significantly larger than 120°, *cf.* 6b,10b-dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene (Hazell & Weigelt, 1976). Therefore, it appears that there is a slight distortion of the dibutylacenaphthofluoranthene molecule.

The title compound is almost symmetrical about the line through the C(11)–C(17) bond. Thus, the molecule is slightly non-planar. Bond lengths and angles suggest that all bonds in the molecule, except the butyl group, are in resonance.

References

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.

HAZELL, A. C., JONES, D. W. & SOWDEN, J. M. (1977). *Acta Cryst.* B33, 1516–1522.

HAZELL, A. C. & WEIGELT, Å. (1976). *Acta Cryst.* B32, 306–308.

HOWELLS, E. R., PHILIPS, D. C. & ROGERS, D. (1950). *Acta Cryst.* 3, 210–214.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–73. Birmingham: Kynoch Press.