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The Crystal Structure of Fluoranthene, $C_{16}H_{10}$: A Study by X-ray and Neutron Diffraction

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The non-alternant hydrocarbon fluoranthene, $C_{16}H_{10}$, is monoclinic, space group $P2_1/n$, with a = 19·907 (18), b = 6·211 (5), c = 18·349 (14) Å, β = 109·86 (8)°, Z = 8. Least-squares refinement gave R = 0·055 for 366 parameters and 2110 reflexions [$I > 3\sigma(I)$] from X-ray diffraction, and R = 0·052 for 469 parameters and 1156 reflexions [$I > 3\sigma(I)$] from neutron diffraction data. The e.s.d.'s of C positions were 0·003 and 0·008 Å for the X-ray and neutron refinements respectively. The two independent molecules in the asymmetric unit deviate slightly from planarity. Their bond lengths correspond roughly to those of a naphthalene moiety linked by single bonds to a benzene ring, and they agree well with the results of MO calculations, except for three bonds involving an atom of the central ring.

Introduction

Although the structures of many aromatic hydrocarbons have been studied, both experimentally and theoretically, almost all the experimental results are for alternant hydrocarbons. For those few non-alternant compounds studied, disorder has generally prevented accurate determination of molecular geometry. To obtain reasonably accurate structural data for a medium-sized non-alternant hydrocarbon, we have determined the structure of fluoranthene by both X-ray and neutron diffraction. The degrees of planarity and of molecular overcrowding in fluoranthene are of interest in connexion with the carcinogenic activity of derivatives (Jones & Matthews, 1974) and with ring-current and other theoretical calculations (Mallion, 1970).

Crystal data

Fluoranthene, $C_{16}H_{10}$, M_r = 202·2. Monoclinic, a = 19·907 (18), b = 6·211 (5), c = 18·349 (14) Å, β = 109·86 (8)°, U = 2133·8 Å^3, D_m = 1·24 (1), D_c = 1·26 g cm^{-3}, Z = 8. With systematic absences $0k0$ with k odd, and $h + l$ with l odd, the space group is $P2_1/n$. The compound crystallizes from benzene as colourless prisms elongated in the [010] direction and terminated by (012). The most prominent prism face is (100): other prism faces present are (001), (101), (10$\bar{1}$) and (10$\bar{3}$). For Mo $K\alpha$ radiation, μ = 0·66 cm^{-1}, and for neutrons (λ = 1·075 Å) μ = 1·55 (1) cm^{-1}, *i.e.* the value measured for pyrene (Hazell, Larsen & Lehmann, 1972).

Experimental

X-ray data

Intensities (hkl, k = 0 to 6) were collected on an Arndt & Phillips (1961) linear diffractometer. Graphite-monochromated Mo $K\alpha$ radiation was used in conjunction with a scintillation counter and a pulse-height analyser. Of the 4241 independent reflexions with $\sin\theta/\lambda < 0{\cdot}7$ recorded, 2129 had $I > 2\sigma_c(I)$, where $\sigma_c(I)$ is the standard deviation from the counting statistics.

The crystal, which was mounted along **b**, was 0·72 mm long and 0·37 × 0·43 mm in cross-section. No corrections were made for absorption.

Neutron data

Intensities were collected on a Hilger–Ferranti four-circle diffractometer at the DR3 reactor of the Danish Atomic Energy Commission Research Establishment, Risø. The wavelength of the monochromatic neutron beam was 1·075 Å. Reflexions were measured in the symmetrical A setting (Furnas & Harker, 1955) with **b** parallel to the φ axis. The ω–2θ scan technique was used and the counts for each step were recorded: the scan width was 2·4 + tan θ, and the step length 0·04°.

For $\sin\theta/\lambda < 0{\cdot}3$, all four symmetry-related reflexions were measured: for $0{\cdot}3 < \sin\theta/\lambda < 0{\cdot}75$, hkl and $h\bar{k}l$ were measured for reflexions for which $I_c > 15$ [I_c(max) = 6972]. The standard reflexions were $\bar{2}04$, measured every 12 reflexions, and 432, measured every 18 reflexions. Since fluoranthene has two relatively

long axes, there were problems in resolving the individual reflexions. The intensities were processed with a modified form of the Lehmann & Larsen (1974) algorithm; in this, the values of $\sigma(I)/I$ enabled the peak limits and also the start of neighbouring peaks to be determined so that the background could be located. Of 1276 independent reflexions, 1160 had $I > 3\sigma_c(I)$. Intensities were corrected for absorption. The crystal, cut from a crystal grown by Dr J. N. Sherwood of the University of Strathclyde, was a block 9·5 × 4·2 × 4·2 mm, elongated along **b**, bounded by (100) and ($\bar{1}03$) and terminated by (010).

Determination and refinement of the structure

The structure was determined from the X-ray data by direct methods (*SYMBAD*, Danielsen, 1969).

X-ray data

Least-squares refinement of atomic coordinates, thermal parameters (anisotropic for C, isotropic for H), a scale factor, and an isotropic extinction factor, g, gave $R = 0{\cdot}055$ and $R_w = 0{\cdot}056$ for 366 parameters and 2110 reflexions. Reflexions for which $I < 3\sigma_c(I)$ were omitted, as were those which were close to the rotation axis, *i.e.* $(h^2 + l^2) < 3$. With $g = 6{\cdot}82 \times 10^{-6}$, the maximum correction for extinction was for $20\bar{4}$, for which $F/F_{\rm corr} = 0{\cdot}52$.

Neutron data

Least-squares refinement of coordinates, anisotropic temperature factors and a scale factor gave $R = 0{\cdot}052$ and $R_w = 0{\cdot}054$ for 469 parameters and 1156 reflexions with $I > 3\sigma_c(I)$. Attempts to refine g always yielded a negative value.

In view of the low ratio of observables to parameters, several constrained refinements were car-

Table 1. *Summary of refinements with neutron data*

For the constrained refinements A, B, and C the thermal motion is described by **T**, **L**, **S** and three extra parameters to account for the internal-mode vibrations of the H atoms. These are compared with a conventional refinement, D, in which individual anisotropic temperature factors are refined. N_o is the number of reflexions, N_p the number of parameters, N_c the number of parameters refined, and $\mathscr{R}_{1,2} = R_w(1)/R_w(2)$.

	N_o	N_c	R	R_w
(*A*) Coordinates unconstrained	1157	204	0·061	0·068
(*B*) Identical molecules, *mm*2	1157	82	0·076	0·088
(*C*) Identical molecules, non-planar	1157	132	0·074	0·085
(*D*) Conventional refinement	1156	469	0·052	0·054

Refinements				Probability levels			
1	2	N_c	N_p	0·05	0·01	0·001	$\mathscr{R}_{1,2}$
A	*D*	204	469	1·206	1·219	1·235	1·259
B	*D*	82	469	1·285	1·300	1·319	1·630
C	*D*	132	469	1·253	1·268	1·285	1·556
B	*C*	82	132	1·033	1·037	1·042	1·048

Table 2. *Atomic coordinates and thermal parameters*

The upper half of the table contains the values from X-ray diffraction, the lower half those from neutron diffraction. For the neutron results and for the C atoms of the X-ray results, coordinates are $\times 10^4$ and U_{ij} are in Å^2 × 10^{-3}; for the X-ray results the coordinates of the H atoms are $\times 10^3$ and the isotropic temperature factor coefficients are in Å^2 × 10^{-3}.

	X	Y	Z	U11	U22	U33	U12	U13	U23
C(1)	1709(2)	757(5)	1143(2)	60(2)	76(2)	40(2)	6(2)	10(2)	4(2)
C(2)	2174(2)	-458(5)	877(2)	95(3)	65(2)	40(2)	20(2)	22(2)	4(2)
C(3)	1937(3)	-2264(7)	461(2)	131(4)	87(3)	66(3)	23(3)	31(3)	-1(2)
C(4)	1162(4)	-2814(8)	318(3)	148(5)	88(3)	68(3)	-14(4)	12(3)	-11(3)
C(5)	744(3)	-1653(7)	591(2)	87(4)	108(3)	69(3)	-22(3)	2(3)	8(3)
C(6)	1001(2)	235(6)	1028(2)	72(3)	85(3)	48(2)	-3(2)	6(2)	5(2)
C(7)	627(2)	1672(7)	1350(2)	57(3)	126(3)	76(3)	6(3)	16(2)	12(3)
C(8)	9650(2)	3441(7)	1751(2)	78(3)	110(3)	80(3)	28(3)	35(2)	4(2)
C(9)	1680(2)	3927(6)	1851(2)	77(3)	86(2)	61(2)	8(3)	20(2)	-7(3)
C(10)	2055(2)	2582(5)	1546(2)	57(2)	77(2)	46(2)	8(2)	15(2)	0(2)
C(11)	2793(2)	2542(5)	1523(2)	60(2)	82(2)	44(2)	10(2)	16(2)	11(2)
C(12)	3356(2)	3918(7)	1817(2)	71(3)	100(3)	73(3)	1(3)	15(2)	12(2)
C(13)	3989(2)	3458(10)	1716(3)	69(3)	143(4)	106(4)	6(3)	27(3)	34(3)
C(14)	4066(3)	1658(10)	1322(3)	75(3)	160(4)	112(4)	43(4)	50(3)	60(4)
C(15)	3506(3)	221(9)	1024(3)	99(4)	110(3)	73(3)	39(3)	44(3)	25(3)
C(16)	2862(2)	677(6)	1122(2)	68(3)	87(2)	46(2)	18(2)	22(2)	18(2)
C(17)	1790(2)	6077(4)	8377(2)	63(2)	62(2)	50(2)	5(2)	27(2)	-4(2)
C(18)	2099(2)	7925(5)	8180(2)	64(2)	65(2)	49(2)	8(2)	27(2)	4(2)
C(19)	1672(2)	9249(5)	7623(2)	79(3)	75(2)	67(2)	9(2)	34(2)	14(2)
C(20)	945(2)	8727(6)	7283(2)	75(3)	90(2)	67(2)	26(2)	22(2)	11(2)
C(21)	644(2)	6959(6)	7493(2)	58(2)	95(2)	70(2)	8(2)	23(2)	-9(2)
C(22)	1067(2)	5536(5)	8057(2)	66(2)	68(2)	62(2)	4(2)	29(2)	-7(2)
C(23)	853(2)	3651(5)	8351(2)	75(3)	79(2)	89(3)	-7(2)	38(2)	-14(2)
C(24)	1346(2)	2467(6)	8908(2)	99(3)	68(2)	93(3)	-11(2)	48(3)	-2(2)
C(25)	2070(2)	3055(5)	9207(2)	96(3)	70(2)	62(2)	14(2)	29(2)	10(2)
C(26)	2300(2)	4852(4)	8946(2)	74(2)	57(2)	51(2)	7(2)	29(2)	1(1)
C(27)	2983(2)	6010(5)	9129(2)	65(2)	72(2)	43(2)	11(2)	20(2)	-1(1)
C(28)	3659(2)	5574(6)	9629(2)	79(3)	84(2)	68(2)	18(2)	19(2)	0(2)
C(29)	4208(2)	7007(7)	9682(2)	64(3)	112(3)	84(3)	15(3)	16(2)	-13(3)
C(30)	4081(2)	8843(7)	9237(2)	70(3)	103(3)	96(3)	-7(3)	36(3)	-14(3)
C(31)	3405(2)	9291(6)	8730(2)	71(3)	87(2)	80(3)	5(2)	37(2)	3(2)
C(32)	2856(2)	7886(5)	8570(2)	59(2)	72(2)	54(2)	2(2)	28(2)	-1(2)

	X	Y	Z	UISO		X	Y	Z	UISO
H(3)	222(2)	-301(5)	31(2)	99(14)	H(19)	189(1)	1045(4)	748(2)	76(9)
H(4)	100(2)	-402(6)	3(2)	146(15)	H(20)	67(1)	972(4)	691(2)	87(10)
H(5)	28(2)	-200(5)	55(2)	83(12)	H(21)	13(1)	663(4)	728(1)	79(10)
H(7)	13(1)	130(4)	127(2)	84(9)	H(23)	36(2)	320(4)	812(2)	95(11)
H(8)	69(1)	435(4)	202(2)	97(9)	H(24)	118(1)	118(4)	911(2)	93(10)
H(9)	191(2)	519(5)	216(2)	106(12)	H(25)	240(1)	221(4)	957(1)	63(8)
H(12)	329(2)	516(5)	211(2)	101(13)	H(28)	373(1)	430(4)	995(1)	65(8)
H(13)	443(2)	435(5)	193(2)	148(14)	H(29)	467(2)	667(5)	1002(2)	116(13)
H(14)	447(2)	123(5)	122(2)	121(13)	H(30)	447(1)	983(4)	927(2)	83(10)
H(15)	352(2)	-105(5)	75(2)	84(13)	H(31)	332(1)	1048(4)	840(2)	78(10)

	X	Y	Z	U11	U22	U33	U12	U13	U23
C(1)	1720(3)	741(11)	1147(3)	71(5)	77(6)	48(4)	-2(5)	15(4)	-1(4)
C(2)	2163(4)	-472(11)	871(4)	91(6)	68(6)	57(5)	21(5)	27(4)	3(4)
C(3)	1920(5)	-2274(12)	452(4)	145(8)	54(5)	63(5)	5(5)	21(5)	-6(4)
C(4)	1179(6)	-2838(13)	300(5)	171(9)	66(6)	81(6)	-16(7)	20(6)	-16(5)
C(5)	743(5)	-1632(14)	602(4)	103(6)	99(7)	84(6)	-28(6)	-3(5)	-4(5)
C(6)	993(3)	221(13)	1026(4)	64(5)	111(7)	64(5)	-6(5)	13(4)	-4(5)
C(7)	630(3)	1657(15)	1363(4)	56(4)	128(8)	97(6)	9(5)	26(4)	15(6)
C(8)	964(4)	3456(14)	1748(4)	87(6)	105(7)	96(6)	26(6)	50(5)	17(5)
C(9)	1686(4)	3944(12)	1851(4)	79(5)	97(6)	72(5)	26(5)	31(4)	-6(4)
C(10)	2054(3)	2594(12)	1553(3)	56(4)	97(6)	53(4)	8(4)	21(3)	2(4)
C(11)	2787(3)	2521(11)	1514(4)	66(4)	80(5)	67(5)	4(4)	28(3)	10(4)
C(12)	3347(4)	3940(14)	1819(4)	79(5)	96(6)	85(5)	-5(5)	19(4)	14(5)
C(13)	3988(4)	3430(19)	1702(5)	54(5)	169(11)	135(8)	4(6)	28(5)	49(7)
C(14)	4067(4)	1599(18)	1316(5)	74(7)	162(11)	127(9)	38(8)	54(6)	57(8)
C(15)	3496(4)	138(16)	1011(5)	86(6)	131(9)	93(6)	49(7)	55(5)	40(6)
C(16)	2865(4)	664(12)	1120(4)	74(5)	86(6)	60(5)	25(5)	24(4)	12(5)
H(3)	2224(10)	-3187(30)	245(10)	164(15)	123(14)	109(12)	-8(13)	57(11)	11(12)
H(4)	935(11)	-4232(36)	-45(9)	243(24)	169(19)	76(12)	-41(18)	13(13)	-40(13)
H(5)	157(9)	-2104(35)	463(10)	94(13)	201(22)	142(15)	-40(15)	27(12)	-8(15)
H(7)	44(7)	1374(34)	1273(10)	52(10)	206(22)	167(16)	-21(13)	43(10)	-28(15)
H(8)	677(9)	4517(29)	2015(10)	130(16)	132(17)	168(18)	40(14)	95(14)	-4(14)
H(9)	1924(8)	5235(29)	2167(9)	115(16)	133(18)	119(17)	-9(14)	42(13)	-51(14)
H(12)	3305(8)	5399(32)	2127(9)	113(14)	156(23)	98(16)	-12(12)	37(15)	-16(-0)
H(13)	4451(8)	4575(37)	1950(11)	72(12)	211(24)	200(20)	-37(14)	34(12)	-4(18)
H(14)	4592(8)	1217(40)	1275(12)	87(11)	279(25)	206(20)	34(16)	88(13)	81(20)
H(15)	3545(9)	-1210(34)	714(9)	134(13)	190(18)	107(12)	65(13)	66(10)	40(13)
C(17)	1791(3)	6075(11)	8377(3)	74(5)	58(5)	54(4)	4(4)	34(3)	-3(4)
C(18)	2094(3)	7911(10)	8172(3)	66(4)	62(5)	60(4)	7(4)	31(4)	5(4)
C(19)	1678(4)	9294(12)	7529(4)	79(7)	81(7)	69(5)	17(6)	30(5)	22(6)
C(20)	945(4)	8741(13)	7280(4)	72(7)	96(8)	74(6)	21(6)	28(5)	15(5)
C(21)	647(3)	6982(13)	7480(4)	56(5)	97(7)	81(6)	16(5)	20(5)	-7(5)
C(22)	1062(3)	5547(12)	8056(4)	64(5)	82(5)	71(5)	-10(4)	32(4)	-15(5)
C(23)	855(4)	3648(12)	8351(4)	81(5)	77(6)	100(6)	-16(6)	49(5)	-16(5)
C(24)	1340(4)	2428(13)	8924(5)	104(8)	70(7)	99(8)	-13(7)	47(5)	0(6)
C(25)	2072(4)	3021(12)	9220(4)	91(8)	63(6)	93(7)	6(5)	35(6)	16(5)
C(26)	2295(3)	4821(11)	8955(3)	75(5)	67(5)	59(4)	16(4)	27(4)	3(4)
C(27)	2984(3)	5988(11)	9130(3)	64(4)	86(6)	58(4)	6(4)	27(3)	7(7)
C(28)	3664(4)	5503(16)	9644(4)	77(6)	78(7)	69(6)	14(6)	20(5)	-10(5)
C(29)	4203(4)	7016(16)	9680(4)	67(5)	107(7)	100(6)	20(6)	15(5)	-7(6)
C(30)	4089(4)	8837(14)	9244(5)	67(6)	105(8)	109(7)	-5(5)	32(5)	-15(6)
C(31)	3405(3)	9314(13)	8731(4)	69(6)	85(7)	98(7)	3(5)	41(5)	5(5)
C(32)	2867(3)	7860(11)	8675(3)	61(4)	79(5)	64(5)	-3(4)	29(3)	-2(4)
H(19)	1891(8)	10714(28)	7464(9)	110(14)	124(17)	105(15)	3(13)	34(11)	36(13)
H(20)	609(8)	9774(26)	6837(9)	105(13)	114(15)	118(14)	27(12)	13(11)	25(13)
H(21)	70(7)	6547(29)	7186(9)	64(10)	151(17)	122(13)	8(12)	3(9)	-24(13)
H(23)	285(7)	3184(25)	8109(9)	82(11)	111(13)	146(13)	-36(11)	47(10)	-23(10)
H(24)	1169(9)	1076(32)	9125(10)	116(15)	134(19)	131(15)	9(15)	41(12)	15(14)
H(25)	2442(9)	2061(27)	9656(9)	132(-0)	123(-0)	90(-0)	-11(-0)	34(-0)	26(-0)
H(25)	3766(9)	4232(27)	9984(9)	93(15)	164(16)	83(12)	16(14)	7(10)	1(12)
H(28)	4762(8)	6720(31)	10093(8)	58(12)	179(21)	140(13)	7(15)	-13(10)	-47(13)
H(29)	4547(7)	9910(31)	9297(10)	73(10)	180(19)	159(14)	-29(12)	49(9)	-45(14)
H(30)	3306(8)	10794(33)	8366(10)	105(11)	118(21)	107(17)	-13(13)	50(11)	19(16)

Table 3. **T**, **L**, *and* **S** *relative to orthogonal axes such that X* ∥ *a, Z* ∥ *c* and Y perpendicular to X and Z*; **T'**, **L'** *and* **S'** *are relative to molecular axes, X' along the molecule and Z' perpendicular to the molecule*

The subscripts refer to molecule 1 or 2; *X* and *N* mean X-ray and neutron diffraction. **T** is in Å^2 × 10^{-4}, **L** in degrees^2, **S** in degrees Å × 10^{-3}. The r.m.s. values of $|U_{ij}$ (obs) − U_{ij} (calc)| are $\Delta_1(X)$ = 0·0045, $\Delta_1(N)$ = 0·0067, $\Delta_2(X)$ = 0·0031, $\Delta_2(N)$ = 0·0046 Å^2 for C atoms only.

	M_{11}	M_{22}	M_{33}	M_{12}	M_{13}	M_{21}	M_{23}	M_{31}	M_{32}
$T_1(X)$	674 (14)	706 (18)	358 (23)	120 (13)	19 (14)		76 (19)		
$T_1(N)$	649 (21)	700 (27)	507 (34)	114 (20)	29 (21)		2 (28)		
$T_1'(X)$	735	617	386	−95	−78		104		
$T_1'(N)$	708	588	559	−95	−56		72		
$T_2(X)$	562 (11)	622 (12)	457 (14)	93 (10)	121 (11)		−1 (12)		
$T_2(N)$	568 (16)	654 (19)	554 (21)	64 (16)	105 (17)		16 (18)		
$T_2'(X)$	676	568	398	44	−62		−70		
$T_2'(N)$	677	604	495	19	−81		−55		
$L_1(X)$	18·8 (2·3)	14·8 (1·0)	18·3 (0·9)	1·2 (1·0)	0·5 (1·0)		−6·8 (0·9)		
$L_1(N)$	9·8 (3·4)	17·2 (1·5)	21·2 (1·3)	−1·4 (1·4)	−1·4 (1·7)		−10·6 (1·3)		
$L_1'(X)$	18·7	9·7	23·3	1·4	−0·6		1·2		
$L_1'(N)$	8·7	9·9	29·7	1·9	−1·3		2·6		
$L_2(X)$	15·6 (1·5)	11·9 (0·8)	10·6 (0·8)	−0·7 (0·7)	2·5 (0·8)		3·8 (0·6)		
$L_2(N)$	16·7 (2·2)	11·6 (1·1)	10·1 (1·2)	−2·0 (1·0)	3·1 (1·2)		4·0 (0·9)		
$L_2'(X)$	16·8	7·8	13·6	−0·7	−0·4		−2·5		
$L_2'(N)$	17·5	7·1	18·9	−1·7	0·3		−3·2		
$S_1(X)$	−1 (52)	−116 (34)	[117 (288)]	−39 (39)	38 (30)	−223 (32)	−68 (18)	240 (25)	52 (17)
$S_1(N)$	35 (77)	−166 (52)	[131 (433)]	−143 (58)	23 (45)	−239 (47)	−51 (28)	297 (37)	59 (26)
$S_1'(X)$	−27	−34	63	23	62	72	−24	354	63
$S_1'(N)$	−16	−31	47	128	113	64	−51	416	47
$S_2(X)$	65 (33)	−49 (27)	[−16 (197)]	89 (25)	20 (21)	97 (17)	14 (14)	106 (17)	105 (16)
$S_2(N)$	52 (49)	−45 (40)	[−7 (297)]	132 (36)	−1 (31)	115 (26)	−11 (32)	116 (25)	97 (24)
$S_2'(X)$	142	−91	−51	84	−8	2	45	−80	−16
$S_2'(N)$	139	−67	−72	126	−14	0	45	−95	4

Table 4. *Extra vibrations,* ΔU (Å^2), *of* H *atoms as a result of internal modes*

ΔU_1 is along the bond, ΔU_3 is perpendicular to the molecular axis, and axis 2 is perpendicular to axes 1 and 3.

	Fluoranthene			
	Molecule 1	Molecule 2	Mean	Pyrene
ΔU_1	0·005 (4)	0·006 (4)	0·005 (3)	0·008 (2)
ΔU_2	0·011 (5)	0·013 (5)	0·012 (4)	0·013 (3)
ΔU_3	0·041 (6)	0·034 (5)	0·038 (4)	0·028 (2)

ried out. The results of these, summarized in Table 1, show the unconstrained neutron refinement to be significantly better than any of the constrained ones. Atomic coordinates and thermal parameters are listed in Table 2.†

The thermal motion of the atoms was analysed on the assumption that the molecule could be treated as a rigid body (Schomaker & Trueblood, 1968). **T**, **L** and **S**, for both X-ray and neutron refinements, are given in Table 3. The values of **L** enabled the bond lengths to be corrected for thermal motion. In the constrained refinements, three additional parameters were included to correct for the neglect of internal modes of vibration: in Table 4 these are compared with the corresponding mean values for pyrene.

Intramolecular bond lengths and angles for the two molecules from the X-ray and neutron refinements are given in Tables 5 and 6: the atoms are numbered and labelled in accordance with Fig. 1. Short Intermolecular distances are listed in Table 7.

† Lists of X-ray and neutron structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32280 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Computational details

Calculations were carried out on a CDC6400 computer with the following programs: data reduction, *PROCH* and *DSORTH* (State University of New York at Buffalo); full-matrix least-squares refinement, *LINUS* (Coppens & Hamilton, 1970); constrained refinements, *KONSLS* (Pawley, 1971); distances and angles, *ORFFE* (Busing, Martin & Levy, 1964); drawings, *ORTEP* (Johnson, 1965); and energy minimization, *STRAIN* (Hazell, 1976).

The quantity minimized was $r = \Sigma w(|F_o| - |F_c|)^2 / \Sigma w|F_o|^2$, where $w = \{[\sigma(F_o^2) + 1{\cdot}03\,F_o^2]^{1/2} - |F_o|\}^{-2}$. The scattering factors were those of Cromer & Mann (1968) for C and of Stewart, Davidson & Simpson (1965) for H; the scattering lengths were those recommended by the Neutron Diffraction Commission

(1972). $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$. The atom–atom potentials were those of Dashevski, Struchkov & Akopyan (1966), and the elastic constants those of Dashevski & Kitaigorodsky (1967).

Discussion

There are two independent molecules in the asymmetric unit (Fig. 2). From magnetic anisotropy measurements, Lasheen (1964) found that $\sin^2\alpha_1 + \sin^2\alpha_2 = 0{\cdot}614$, where α_1 and α_2 are the angles between the normals to the molecules and (010), and that the magnetic susceptibility χ_2 was in (010) at 9° to **a** in obtuse β. [The Chakravarti (1954) axes in space group $P2_1/c$ used by Lasheen have been transformed to those of our cell.] From the present structure determination, $\alpha_1 = 31{\cdot}9°$ and $\alpha_2 = 31{\cdot}6°$, so that $\sin^2\alpha_1 + \sin^2\alpha_2 = 0{\cdot}554$; the projections of the normals on (010) make angles of 8·5 and 24·6° with **a** in obtuse β. The molecules are not identical and are slightly non-planar [see Table 8 and *cf.* pyrene and ovalene (Hazell & Pawley, 1973)].

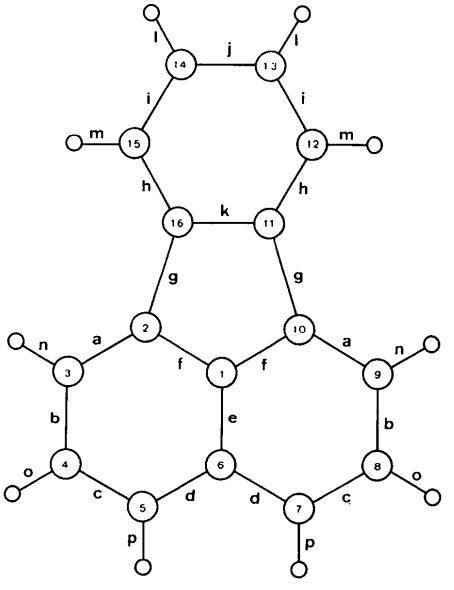

Fig. 1. Molecule 1 of fluoranthene showing the numbering of atoms and the labelling of bonds. The H atoms, represented by small circles, have the same numbers as the C atoms to which they are attached. The numbering of molecule 2 is such that atom *j* in molecule 2 corresponds to atom *j* − 16 in molecule 1.

For aromatic hydrocarbons [*e.g.* pyrene (Hazell, Larsen & Lehmann, 1972), triphenylene (Ferraris, Jones & Yerkess, 1973), and phenanthrene (Kay, Okaya & Cox, 1971; Jones & Yerkess, 1971)], bond lengths, l_X, determined by X-ray diffraction may be systematically in error because of the asphericity of the electron clouds: short bonds appear shorter than l_{calc} and long bonds longer. While the same effect is observed with fluoranthene, the bond lengths, l_N, from neutron diffraction do not here agree well with the theoretical values: $\Delta(l_N - l_X)$ sometimes has the opposite sign to $\Delta(l_{calc} - l_X)$. The results from the neutron diffraction study are only moderately accurate [$\langle\sigma_N(C)\rangle = 0{\cdot}008$ Å, *cf.* $\langle\sigma_X(C)\rangle = 0{\cdot}003$ Å]; moreover, the consistently negative values obtained when attempts were made to refine an extinction parameter, *g*, suggest that there may be some systematic errors in the neutron data. These could arise from the use of too large a crystal and from inadequate resolving power in the diffractometer for a unit cell with two long axes. Errors in either or both of the *X* and *N* sets of data are indicated by the poor agreement of some **L** and **S** (Table 3), *e.g.* L_{11} for molecule 1, and by the differences in the deviations of the molecules from planarity (Table 8). The neutron diffraction data, however, do lead to a

Table 5. *Bond lengths* (Å)

	X-ray				Neutron			
	Molecule 1		Molecule 2		Molecule 1		Molecule 2	
a	1·372 (4)	1·367 (3)	1·367 (4)	1·360 (4)	1·360 (8)	1·354 (8)	1·368 (8)	1·361 (8)
b	1·407 (5)	1·413 (4)	1·411 (4)	1·411 (4)	1·458 (11)	1·424 (10)	1·426 (8)	1·424 (10)
c	1·360 (5)	1·374 (4)	1·366 (4)	1·371 (4)	1·405 (11)	1·376 (10)	1·259 (10)	1·393 (10)
d	1·423 (4)	1·423 (4)	1·420 (4)	1·420 (4)	1·391 (10)	1·424 (10)	1·421 (8)	1·424 (8)
e	1·397 (4)		1·403 (3)		1·414 (8)		1·412 (8)	
f	1·411 (3)	1·409 (3)	1·411 (3)	1·413 (3)	1·404 (8)	1·424 (8)	1·405 (8)	1·427 (8)
g	1·476 (4)	1·473 (4)	1·475 (4)	1·478 (3)	1·500 (8)	1·494 (8)	1·507 (7)	1·490 (8)
h	1·388 (4)	1·391 (4)	1·379 (4)	1·379 (4)	1·391 (8)	1·385 (8)	1·399 (8)	1·383 (8)
i	1·373 (4)	1·395 (5)	1·391 (4)	1·383 (4)	1·408 (10)	1·423 (12)	1·415 (10)	1·404 (10)
j	1·376 (5)		1·382 (4)		1·384 (13)		1·365 (11)	
k	1·419 (3)		1·415 (3)		1·405 (8)		1·410 (8)	
l	0·93 (3)	0·99 (3)	0·97 (2)	0·94 (3)	1·11 (2)	1·14 (2)	1·11 (2)	1·13 (2)
m	0·95 (2)	0·97 (3)	0·93 (2)	0·97 (3)	1·03 (3)	1·09 (2)	1·12 (2)	0·99 (2)
n	0·91 (2)	0·99 (3)	0·95 (2)	0·02 (2)	1·00 (2)	1·04 (2)	1·07 (2)	1·07 (2)
o	0·92 (2)	1·02 (2)	0·95 (2)	0·99 (3)	1·09 (2)	1·10 (2)	1·08 (2)	1·03 (2)
p	0·94 (2)	0·98 (2)	0·98 (2)	0·07 (2)	1·15 (2)	1·12 (2)	1·11 (2)	1·11 (2)

Table 6. *Bond angles* (°)

	X-ray				Neutron				Mean values		
	Molecule 1		Molecule 2		Molecule 1		Molecule 2		X-ray	Neutron	Calculated
ab	119·8 (3)	119·0 (3)	118·7 (3)	120·0 (3)	120·7 (7)	118·7 (6)	117·0 (6)	119·9 (6)	119·4 (2)	119·1 (3)	118·7
af	116·6 (3)	118·4 (3)	118·1 (3)	117·6 (3)	120·2 (6)	119·2 (5)	120·0 (5)	119·0 (5)	117·7 (2)	119·6 (3)	117·9
bc	122·7 (4)	122·6 (3)	123·3 (3)	122·2 (3)	121·6 (7)	122·5 (6)	123·4 (6)	120·5 (6)	122·7 (2)	122·0 (3)	122·9
cd	120·0 (4)	119·8 (3)	119·9 (3)	120·0 (3)	121·1 (7)	120·6 (5)	120·8 (5)	121·8 (6)	119·9 (2)	121·1 (5)	120·7
de	115·6 (3)	116·2 (3)	115·6 (3)	116·0 (3)	115·6 (6)	115·3 (6)	115·1 (5)	115·3 (6)	115·9 (2)	115·3 (3)	115·4
ef	125·2 (3)	124·0 (3)	124·4 (3)	124·2 (3)	124·5 (6)	123·7 (5)	123·7 (5)	123·5 (5)	124·5 (2)	123·9 (3)	124·5
ff	110·8 (3)		111·4 (2)		111·9 (5)		112·8 (5)		111·1 (2)	112·4 (4)	111·0
fg	107·1 (2)	106·4 (2)	106·1 (2)	106·4 (2)	106·7 (5)	104·9 (5)	105·3 (5)	105·1 (5)	106·5 (1)	105·5 (2)	105·7
gk	107·1 (2)	108·5 (3)	108·4 (2)	107·7 (2)	107·0 (5)	109·5 (5)	108·1 (5)	108·8 (4)	107·9 (1)	108·3 (2)	108·8
hk	120·5 (3)	119·4 (3)	120·0 (3)	119·8 (3)	122·6 (6)	121·3 (5)	122·1 (5)	120·4 (5)	119·9 (2)	121·6 (3)	120·5
hi	118·2 (4)	119·7 (4)	119·7 (3)	119·4 (3)	116·5 (7)	116·2 (7)	117·8 (6)	116·1 (6)	119·2 (2)	116·7 (3)	118·5
ij	121·1 (3)	121·1 (4)	120·5 (3)	120·6 (3)	121·1 (6)	122·3 (7)	120·1 (6)	123·6 (6)	120·8 (2)	121·8 (3)	120·9
ag	136·2 (3)	135·2 (3)	135·9 (3)	136·0 (3)	133·1 (6)	135·9 (6)	134·8 (5)	135·9 (5)	135·8 (2)	134·9 (3)	136·5
dd	128·2 (3)		128·4 (3)		129·1 (6)		129·7 (6)		128·3 (2)	129·4 (4)	129·3
gh	132·3 (3)	132·1 (3)	131·5 (3)	132·5 (3)	130·4 (6)	129·2 (6)	129·8 (5)	130·9 (5)	132·1 (2)	130·1 (3)	130·6
an	115 (2)	121 (2)	117 (2)	119 (1)	123 (1)	121 (1)	122 (1)	120 (1)	118·0 (0·9)	121·5 (0·5)	121·0
bn	125 (2)	120 (2)	124 (2)	121 (1)	120 (1)	120 (1)	121 (1)	120 (1)	122·5 (0·9)	120·3 (0·5)	120·3
bo	119 (2)	119 (2)	115 (2)	120 (2)	122 (1)	118 (1)	119 (1)	120 (1)	118·3 (0·9)	119·8 (0·5)	118·2
co	118 (2)	118 (2)	121 (2)	118 (2)	116 (1)	119 (1)	118 (1)	119 (1)	118·8 (1·0)	118·0 (0·5)	119·0
cp	126 (2)	125 (2)	124 (1)	122 (2)	120 (1)	120 (1)	120 (1)	121 (1)	124·3 (0·9)	120·3 (0·5)	120·0
dp	114 (2)	116 (2)	116 (1)	118 (2)	119 (1)	119 (1)	119 (1)	117 (1)	116·0 (0·9)	118·5 (0·5)	119·3
hm	117 (2)	117 (2)	119 (2)	118 (1)	122 (1)	123 (1)	122 (1)	123 (1)	117·8 (0·9)	122·5 (0·5)	120·8
il	111 (2)	124 (2)	119 (1)	121 (3)	119 (1)	117 (1)	121 (1)	119 (1)	118·8 (1·1)	119·0 (0·5)	119·6
im	125 (2)	123 (2)	121 (2)	122 (1)	122 (1)	120 (1)	121 (1)	121 (1)	122·8 (0·9)	121·0 (0·5)	120·7
jl	128 (2)	115 (2)	121 (1)	122 (2)	119 (1)	121 (1)	119 (1)	118 (1)	121·5 (0·9)	119·3 (0·5)	120·9

Table 7. *Close intermolecular contacts*, C–C<3·5 Å, C–H < 3·0 Å, H–H < 2·5 Å

Symmetry code

(i)	$x, y, z-1$	(vii)	$1-x, 1-y, 2-z$
(ii)	$x, y-1, z$	(viii)	$\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$
(iii)	$x, y-1, z-1$	(ix)	$\frac{1}{2}-x, \frac{1}{2}+y, 1\frac{1}{2}-z$
(iv)	$x, 1+y, z$	(x)	$\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$
(v)	$-x, 1-y, 1-z$	(xi)	$\frac{1}{2}+x, 1\frac{1}{2}-y, z-\frac{1}{2}$
(vi)	$1-x, 1-y, 1-z$		

C(4)–C(9^{ii})	3·342 Å	C(19)–H(31^{ix})	2·846 Å
C(19)–C(24^{iv})	3·312	C(20)–H(31^{ix})	2·859
C(2)–H(25^{i})	2·931	C(25^{iii})–H(3)	2·965
C(7)–H(12^{viii})	2·965	C(26^{iii})–H(3)	2·718
C(8)–H(12^{viii})	2·818	C(27^{iii})–H(3)	2·974
C(9)–H(12^{viii})	2·888	C(28^{iii})–H(15)	2·895
C(10)–H(9^{viii})	2·909	C(29^{iii})–H(15)	2·867
C(11)–H(9^{viii})	2·677	C(32)–H(19^{ix})	2·658
C(14)–H(21^{x})	2·904	H(8)–H(23^{v})	2·337
C(15)–H(25^{i})	2·909	H(13)–H(20^{xi})	2·419
C(16)–H(25^{i})	2·672	H(14)–H(21^{x})	2·404
C(18)–H(19^{ix})	2·986	H(14)–H(30^{vi})	2·387
C(18)–H(31^{ix})	2·967	H(29)–H(29^{vii})	2·408

more precise location of the H atoms: $\langle\sigma_N(H)\rangle = 0{\cdot}018$ Å, *cf.* $\langle\sigma_X(H)\rangle = 0{\cdot}030$ Å. While the X-ray data were insufficient to allow a high-order refinement to be carried out (for comparison with the neutron diffraction results), the mean apparent C–H length of 0·96 Å from the X-ray refinement was significantly shorter than the 1·10 Å derived from the neutron refinement.

In Table 9, the bond lengths are compared with those for the picryl bromide complex (Herbstein & Kaftory, 1975) and with the results of several theoretical calculations. The generally good agreement between the two X-ray studies implies that there is remarkably little distortion of the fluoranthene molecule in the complex. Whereas deviation of fluoranthene from planarity in the complex is approximately symmetrical about the line through bond *e* produced (Fig. 1), deviations from planarity in both molecules in the fluoranthene structure (Table 8) involve chiefly one ring of the

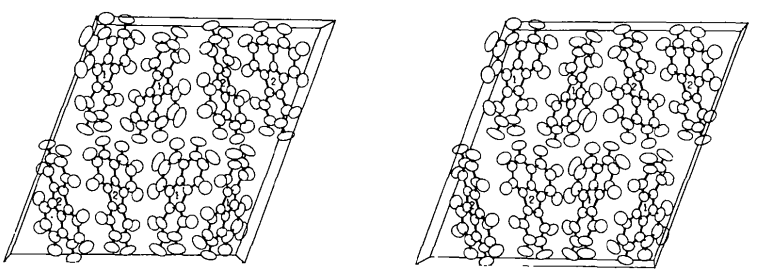

Fig. 2. The unit-cell contents viewed along **b**; **a** is down the page, **c** across the page.

naphthalene moiety and the diametrically opposite region of the benzene moiety. The biggest discrepancies between observed and calculated lengths are for bonds *a* and *f*, which are calculated too long and for *g*, which is calculated too short: each of these bonds involves at least one atom of the five-membered ring.

Table 8. *Deviations from best planes through the carbon atoms (in* Å × 10^{-3})

The mean standard deviations are: $\sigma(C_X) = 0.003$, $\sigma(H_X) = 0.030$, $\sigma(C_N) = 0.008$, $\sigma(H_N) = 0.018$ Å.

	Molecule 1		Molecule 2	
	X	*N*	*X*	*N*
C(1)	−9	−19	−17	−21
C(2)	−23	−11	−4	−17
C(3)	−4	5	−16	2
C(4)	31	50	−18	−21
C(5)	17	9	−6	−10
C(6)	−5	−6	2	6
C(7)	−1	−23	27	30
C(8)	−12	2	33	45
C(9)	−45	3	−4	−1
C(10)	−4	−9	−31	−27
C(11)	−11	17	−20	−28
C(12)	26	31	−41	−45
C(13)	18	28	−9	−5
C(14)	23	−11	33	35
C(15)	−28	−38	46	55
C(16)	16	−18	14	3
H(3)	−37	3	−50	14
H(4)	57	99	−1	−4
H(5)	−33	64	55	−23
H(7)	−3	−23	10	47
H(8)	−102	−37	74	69
H(9)	−40	−22	−42	0
H(12)	11	60	−34	−40
H(13)	−9	35	−43	0
H(14)	10	−82	37	21
H(15)	28	−46	19	−77

High-resolution NMR measurements in solution (Bartle, Jones & Pearson, 1967) led to *ortho* spin–spin coupling constants, J_{ortho}, linearly related (Bartle, Jones & Matthews, 1969) to HMO bond order, *P*, and revealed no detectable inter-ring coupling. Semi-empirical MO calculations (Mallion, 1970) indicate negligible π-electron ring current in the central ring of fluoranthene and its isosteres (Mallion, 1973; Bartle, Jones, Matthews, Birch & Crombie, 1971). For bond *b*, predicted and found to be appreciably longer than *c* in the crystal, J_{ortho} (Bartle, Jones & Pearson, 1967; Heffernan, Jones & Black, 1967) is, as might be expected, over 1 Hz less than J_c. For bonds *i* and *j*, however, predicted to be the same length from bond-order calculations, $l_i > l_j$ is consistently found in the crystal but surprisingly $J_i > J_j$ in several solvents. There is also NMR evidence from dilution shifts (Bartle, Mallion, Jones & Pickles, 1974) that in solution partial parallel alignment of pairs of presumed planar fluoranthene molecules occurs.

The strain introduced by the presence of a five-membered ring between the benzene and naphthalene nuclei is propagated throughout the molecule, so that angle *hi* is smaller than 120° and angle *dd* is significantly larger than 120°, *cf.* 6b,10b-dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene (Hazell & Hazell, 1977). The angles calculated on the assumption of a planar fluoranthene molecule with the expected bond lengths, and with the angles allowed to vary so as to minimize energy, agree well with the observed values (Table 9).

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Table 9. *Observed and calculated bond lengths* (Å): *X by X-ray diffraction, N by neutron diffraction, and P for the picryl bromide complex*

	X	*N*	*P*	*A*	*B*	*C*	*D*	Mean theoretical	Mean observed
a	1·367 (2)	1·361 (5)	1·372 (5)	1·389	1·380	1·390	1·393	1·388	1·367 (2)
b	1·411 (2)	1·433 (6)	1·428 (6)	1·412	1·420	1·416	1·408	1·414	1·414 (2)
c	1·368 (2)	1·383 (6)	1·370 (6)	1·383	1·375	1·384	1·386	1·382	1·370 (2)
d	1·422 (2)	1·415 (5)	1·425 (6)	1·423	1·427	1·425	1·421	1·424	1·421 (2)
e	1·400 (3)	1·413 (6)	1·392 (7)	1·413	1·404	1·415	1·407	1·410	1·401 (3)
f	1·411 (2)	1·415 (5)	1·406 (5)	1·428	1·429	1·428	1·425	1·428	1·411 (2)
g	1·476 (2)	1·498 (4)	1·475 (5)	1·456	1·466	1·464	1·456	1·461	1·480 (2)
h	1·384 (2)	1·390 (5)	1·390 (5)	1·400	1·401	1·404	1·405	1·403	1·385 (2)
i	1·386 (2)	1·413 (6)	1·390 (9)	1·399	1·394	1·398	1·395	1·397	1·389 (2)
j	1·379 (3)	1·375 (9)	1·380 (9)	1·397	1·399	1·402	1·401	1·400	1·379 (3)
k	1·417 (2)	1·408 (6)	1·420 (9)	1·415	1·403	1·410	1·410	1·410	1·416 (2)

(*A*) Warren & Yandle (1968). (*B*) Dewar & Trinajstić (1970). (*C*) and (*D*) Kolc, Thulstrup & Michl (1974).

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The Crystal and Molecular Structure of α-Aminomethylmethylphosphinic Acid

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The structure of α-aminomethylmethylphosphinic acid ($H_3^+N-CH_2-PCH_3O_2^-$) has been determined by single-crystal X-ray diffraction. Crystals are monoclinic, space group $P2_1/c$, with $a = 5{\cdot}867\,(1)$, $b = 6{\cdot}279\,(1)$, $c = 13{\cdot}262\,(2)$ Å, $\beta = 98{\cdot}91\,(1)°$, $Z = 4$. The structure was solved by the heavy-atom technique and refined by full-matrix least squares to $R = 0{\cdot}043$ for 533 counter reflexions for which $F > 3{\cdot}92\,\sigma(F)$. The molecule exists as $H_3^+N-CH_2-PCH_3O_2^-$. The NH_3^+ group is a donor for three hydrogen bonds of lengths 2·775, 2·778, 2·747 Å.

Introduction

Interest in aminophosphinic and aminophosphonic acids stems from the fact that they are P analogues of the amino acids. Many papers deal with aminophosphonic acids but only two report structures (Okaya, 1966; Darriet, Darriet, Cassaigne & Neuzil, 1975).

However, there are no publications dealing with