Structure of 5-Methylchrysene, C$_{19}$H$_{14}$

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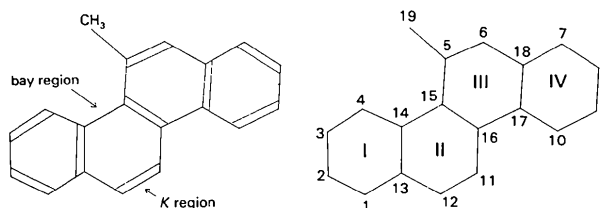*American Health Foundation, Naylor Dana Institute for Disease Prevention, Valhalla, New York* 10595, *USA*

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Abstract. $M_r = 242.32$, $P2_1/c$, $a = 21.680$ (7), $b = 5.558$ (2), $c = 23.876$ (7) Å, $\beta = 116.07$ (2)°, $V = 2584.3$ (1.5) Å^3, $Z = 8$, $D_x = 1.246$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 0.46$ mm^{-1}, $F(000) = 1024$, $T = 298$ K, final $R = 0.080$ for 2636 reflections above $\sigma(I)$. There are two molecules in the asymmetric unit and one of these is disordered, a fact that caused problems in the solution of the structure. The molecules are distorted because of steric hindrance between the 5-methyl group and the hydrogen atom on C(4); this distortion is evidenced by an out-of-plane displacement (0.2–0.4 Å) of the carbon atom of the methyl group and by increased angles (122–127°) in the 'bay region' between C(4) and C(5).

Introduction. 5-Methylchrysene, (I), has been shown to be the only monomethylchrysene that is carcinogenic (Hoffmann, Bondinell & Wynder, 1974); chrysene itself is not. The structure of (I) has been determined in order to investigate the molecular distortions that would be expected as a result of steric hindrance due to the 5-methyl group, that is, between the hydrogen atoms on C(4) and one of the hydrogen atoms on C(19). The crystal structure analysis showed that there was disorder in the packing of one of the two molecules in the asymmetric unit, and this fact unfortunately caused problems in the structure solution and in the precision of the final structure refinement. In spite of these difficulties it has been possible to describe in a quantitative manner the distortion to the chrysene nucleus caused by the 5-methyl group.

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Experimental. Crystals of (I) grown from methanolic solution by slow evaporation; colorless prisms elongated along **b**, crystal 0.09 × 0.45 × 0.19 mm, Syntex $P2_1$ diffractometer, graphite-monochromatized Cu $K\alpha$, lattice parameters from least-squares fit of angular data for 14 reflections, three-dimensional data measured using variable θ–2θ scan, 2θ 0–138° (max. $\sin\theta/\lambda = 0.606$ Å^{-1}), min. scan rate 2° min^{-1}, scan:background ratio 2.0; intensities recorded for 4818 unique data, 2636 with $I \geq 1.0\sigma(I)$ where $\sigma(I)$ is determined from counting statistics, $\sigma(F)$ from $\sigma(F) = (F/2) [\sigma^2(I)/(I)^2 + \delta^2]^{1/2}$ where $\delta(= 0.020)$ is an instrumental uncertainty determined from the variation in intensities of four check reflections periodically monitored during data collection; structure amplitudes obtained by correcting intensities for Lorentz and polarization factors and placed on an absolute scale by a Wilson plot; diffuse scattering evident in Weissenberg photographs $hk0$ through $hk4$ but no attempt made to analyze it for features. Structure solved using *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) with 299 E values ≥ 1.80; resulting E map revealed positions of 41 carbon atoms and indicated that, of the two molecules in the asymmetric unit, one is ordered and the other disordered in two possible orientations; remaining carbon atoms found from Fourier difference maps; structure refined by full-matrix least squares on F, anisotropic, occupancies of 0.5 for disordered atoms; after several refinement cycles, some hydrogen atoms,

including those of methyl groups, located from Fourier difference maps, other hydrogen atoms placed in geometrically idealized positions; totally unconstrained full-matrix least-squares refinement, anisotropic carbons, isotropic hydrogens (Gantzel, Sparks, Long & Trueblood, 1969; Carrell, 1975), $w \propto 1/\sigma^2(F)$, $R = 0.095$ ($wR = 0.092$) for 2636 reflections. Structure subjected to constrained refinement using Oxford *CRYSTALS* program suite (Carruthers & Watkin, 1980); slack constraints (Waser, 1963; Rollett, 1969) applied to bond lengths, interbond angles and thermal parameters of atoms of disordered molecules; carbon atoms refined anisotropically, hydrogen atoms in ordered molecule refined isotropically and only temperature factors of hydrogen atoms of disordered molecules refined; thermal parameter constraint: for two bonded atoms, the components of the thermal ellipsoids in the direction of the bond should be equal within the e.s.d.; refinement used a two-block approximation to the normal matrix (values of x, y, z and B_{ij}) and weights computed from the Chebyshev series: $w = [2079t_0(x) + 2845t_1 \times (x) + 801t_2(x)]$, $x = F_{obs}/F_{max}$; in last two cycles, only constraints were those applied to thermal parameters; refinement converged at $R = 0.080$ ($wR = 0.086$); in the final Fourier difference map $\Delta\rho$ excursions $\leq 0.17\,e\,\text{Å}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); computer programs from the Crystallographic Program Library of the Institute for Cancer Research (Carrell, Shieh & Takusagawa, 1981) and the Oxford *CRYSTALS* program system (Carruthers & Watkin, 1980); also, *MOLCON* (Fujii, 1979), *ORTEP* (Johnson, 1965), *ENER* (Kashino, 1977), *VIEW* (Carrell, 1976) and the *CHEMGRAF* system (Davies, 1983).*

Discussion. The final atomic parameters, from the constrained least-squares refinement ($R = 0.080$), are listed in Table 1. Bond lengths and interbond angles are given in Table 2. Diagrams of the molecular skeleton with atomic numbering and thermal ellipsoids (50% probability) are shown in Fig. 1 (Davies, 1983). These diagrams show that the thermal ellipsoids are larger for the 'ordered' than the disordered molecule. This fact, together with the poor fit to **TLS** data (Carruthers & Watkin, 1980), suggest that there may also be some disorder in this molecule. Short C—C bonds, indicating some localization of double-bond character, are found

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* Lists of structure factors, anisotropic thermal parameters, the parameters of H atoms, intermolecular contacts, torsion angles, and diagrams of bond lengths and angles of molecules have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38987 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic parameters*

This table contains parameters from the constrained least-squares refinement. For each atom positional parameters are listed as fractions of cell edges. Temperature factors have the units of Å². E.s.d.'s for positional parameters are given in parentheses for the last digits quoted and those for temperature factors, which are averages of $U(11)$, $U(22)$ and $U(33)$, are given for each $U(ii)$ in the deposited tables but are of the order of 0.005 Å².

	x	y	z	U_{eq}
C(1)	0.3587 (3)	0.1980 (11)	0.5417 (3)	0.142
C(2)	0.3383 (3)	0.0479 (12)	0.4939 (3)	0.149
C(3)	0.3814 (4)	−0.1303 (13)	0.4957 (3)	0.169
C(4)	0.4451 (3)	−0.1537 (11)	0.5439 (3)	0.152
C(5)	0.5907 (2)	−0.1754 (8)	0.6531 (2)	0.109
C(6)	0.6497 (3)	−0.1733 (9)	0.7050 (3)	0.122
C(7)	0.7301 (3)	−0.0198 (13)	0.8101 (3)	0.136
C(8)	0.7458 (3)	0.1416 (16)	0.8566 (3)	0.141
C(9)	0.6974 (4)	0.3230 (12)	0.8507 (3)	0.139
C(10)	0.6366 (3)	0.3282 (10)	0.8000 (3)	0.125
C(11)	0.5049 (3)	0.3395 (9)	0.6919 (2)	0.114
C(12)	0.4437 (3)	0.3534 (10)	0.6425 (3)	0.129
C(13)	0.4232 (2)	0.1881 (8)	0.5909 (2)	0.113
C(14)	0.4707 (2)	0.0071 (7)	0.5939 (2)	0.098
C(15)	0.5381 (2)	0.0002 (7)	0.6476 (2)	0.096
C(16)	0.5527 (2)	0.1652 (8)	0.6950 (2)	0.100
C(17)	0.6187 (2)	0.1622 (8)	0.7510 (2)	0.101
C(18)	0.6666 (3)	−0.0105 (9)	0.7569 (2)	0.110
C(19)	0.5854 (3)	−0.3540 (10)	0.6049 (3)	0.138
C(41)	0.1350 (6)	0.3689 (24)	0.9549 (7)	0.111
C(42)	0.1734 (11)	0.2049 (31)	0.9966 (10)	0.136
C(43)	0.1970 (8)	0.0065 (29)	0.9787 (6)	0.119
C(44)	0.1868 (5)	−0.0093 (19)	0.9189 (4)	0.097
C(45)	0.1743 (4)	−0.0256 (16)	0.7847 (3)	0.080
C(46)	0.1565 (4)	−0.0326 (16)	0.7235 (4)	0.092
C(47)	0.0920 (7)	0.1100 (19)	0.6144 (6)	0.112
C(48)	0.0461 (13)	0.2698 (31)	0.5727 (13)	0.130
C(49)	0.0161 (7)	0.4399 (24)	0.5945 (8)	0.123
C(410)	0.0333 (6)	0.4615 (20)	0.6563 (5)	0.106
C(411)	0.0705 (6)	0.5092 (26)	0.7880 (5)	0.092
C(412)	0.0821 (4)	0.5246 (16)	0.8485 (5)	0.099
C(413)	0.1240 (4)	0.3557 (16)	0.8925 (5)	0.088
C(414)	0.1520 (4)	0.1633 (17)	0.8729 (4)	0.080
C(415)	0.1420 (4)	0.1522 (14)	0.8078 (4)	0.075
C(416)	0.0967 (4)	0.3156 (14)	0.7661 (4)	0.077
C(417)	0.0797 (4)	0.3044 (17)	0.7000 (4)	0.081
C(418)	0.1071 (5)	0.1217 (16)	0.6788 (5)	0.088
C(419)	0.2317 (4)	−0.1947 (16)	0.8247 (4)	0.098
C(21)	0.0392 (5)	0.3921 (22)	0.6081 (5)	0.097
C(22)	0.0502 (9)	0.2159 (27)	0.5737 (9)	0.094
C(23)	0.0897 (6)	0.0221 (19)	0.6033 (6)	0.096
C(24)	0.1188 (5)	0.0061 (18)	0.6668 (4)	0.088
C(25)	0.1836 (4)	−0.0248 (15)	0.8108 (3)	0.072
C(26)	0.1977 (4)	−0.0420 (14)	0.8719 (4)	0.086
C(27)	0.1861 (5)	0.0792 (18)	0.9660 (5)	0.091
C(28)	0.1647 (6)	0.2431 (30)	0.9970 (7)	0.089
C(29)	0.1234 (8)	0.4302 (24)	0.9634 (6)	0.098
C(210)	0.1056 (5)	0.4617 (17)	0.9015 (5)	0.089
C(211)	0.0636 (6)	0.5040 (23)	0.7678 (5)	0.080
C(212)	0.0451 (4)	0.5392 (15)	0.7079 (4)	0.087
C(213)	0.0641 (4)	0.3690 (16)	0.6732 (4)	0.078
C(214)	0.1079 (5)	0.1771 (17)	0.7052 (4)	0.079
C(215)	0.1330 (3)	0.1527 (13)	0.7725 (3)	0.065
C(216)	0.1094 (4)	0.3224 (14)	0.8024 (4)	0.073
C(217)	0.1274 (4)	0.2979 (16)	0.8686 (4)	0.075
C(218)	0.1700 (4)	0.1138 (17)	0.9023 (4)	0.081
C(219)	0.2208 (4)	−0.2008 (15)	0.7881 (4)	0.088

for C(1)—C(2), C(3)—C(4), C(5)—C(6), C(7)—C(8), C(9)—C(10) and C(11)—C(12), as expected.

This analysis has shown that each molecule is approximately planar apart from the methyl group C(19) attached to C(5). Considerable distortions were expected because of steric interaction between hydrogen atoms on C(19), C(4) and C(6). Data on the planarity of the three molecules, including dihedral angles between rings, are given in Table 3. The largest dihedral angles are seen between rings I and III, that is,

Table 2. Bond lengths (Å) and angles (°)

	Ordered molecule	Disordered molecules	
		Molecule B	Molecule C
C(1)–C(2)	1·323 (7)	1·34 (2)	1·37 (2)
C(1)–C(13)	1·378 (6)	1·40 (2)	1·41 (1)
C(2)–C(3)	1·349 (7)	1·36 (2)	1·36 (2)
C(3)–C(4)	1·362 (7)	1·35 (2)	1·37 (2)
C(4)–C(14)	1·396 (6)	1·40 (1)	1·41 (1)
C(5)–C(6)	1·333 (6)	1·34 (1)	1·36 (1)
C(5)–C(15)	1·462 (5)	1·45 (1)	1·46 (1)
C(5)–C(19)	1·487 (6)	1·52 (1)	1·51 (1)
C(6)–C(18)	1·446 (7)	1·42 (1)	1·42 (1)
C(7)–C(8)	1·348 (8)	1·38 (3)	1·38 (2)
C(7)–C(18)	1·405 (7)	1·43 (1)	1·42 (1)
C(8)–C(9)	1·417 (8)	1·37 (2)	1·38 (2)
C(9)–C(10)	1·343 (7)	1·36 (2)	1·36 (2)
C(10)–C(17)	1·403 (6)	1·39 (1)	1·41 (1)
C(11)–C(12)	1·335 (6)	1·36 (1)	1·32 (1)
C(11)–C(16)	1·398 (6)	1·42 (2)	1·40 (2)
C(12)–C(13)	1·441 (6)	1·40 (1)	1·43 (1)
C(13)–C(14)	1·419 (5)	1·41 (1)	1·41 (1)
C(14)–C(15)	1·461 (5)	1·47 (1)	1·46 (1)
C(15)–C(16)	1·381 (5)	1·39 (1)	1·41 (1)
C(16)–C(17)	1·467 (6)	1·46 (1)	1·46 (1)
C(17)–C(18)	1·376 (6)	1·38 (1)	1·38 (1)
C(2)–C(1)–C(13)	122·8 (6)	120·9 (11)	121·0 (10)
C(1)–C(2)–C(3)	118·0 (6)	121·1 (19)	119·4 (15)
C(2)–C(3)–C(4)	121·9 (6)	118·4 (14)	120·4 (12)
C(3)–C(4)–C(14)	122·4 (6)	124·4 (11)	123·4 (9)
C(6)–C(5)–C(15)	118·0 (5)	118·6 (8)	118·3 (8)
C(6)–C(5)–C(19)	116·2 (5)	115·7 (8)	115·8 (8)
C(15)–C(5)–C(19)	125·7 (5)	125·6 (7)	125·9 (7)
C(5)–C(6)–C(18)	125·4 (5)	124·3 (9)	124·1 (8)
C(8)–C(7)–C(18)	120·9 (6)	120·0 (13)	120·4 (10)
C(7)–C(8)–C(9)	118·9 (6)	118·8 (22)	118·2 (14)
C(8)–C(9)–C(10)	119·8 (6)	121·5 (16)	122·0 (10)
C(9)–C(10)–C(17)	122·6 (6)	121·4 (11)	121·0 (11)
C(12)–C(11)–C(16)	120·8 (5)	121·6 (10)	123·0 (10)
C(11)–C(12)–C(13)	122·1 (5)	120·7 (10)	120·2 (9)
C(1)–C(13)–C(12)	121·3 (5)	120·8 (10)	120·7 (9)
C(1)–C(13)–C(14)	120·9 (5)	119·7 (10)	120·6 (9)
C(12)–C(13)–C(14)	117·8 (5)	119·4 (10)	118·8 (9)
C(4)–C(14)–C(13)	113·9 (5)	115·0 (8)	115·0 (8)
C(4)–C(14)–C(15)	127·0 (5)	125·2 (8)	124·4 (8)
C(13)–C(14)–C(15)	119·1 (4)	119·9 (9)	120·4 (9)
C(5)–C(15)–C(14)	122·5 (4)	123·9 (8)	124·5 (7)
C(5)–C(15)–C(16)	118·7 (5)	118·7 (7)	118·4 (7)
C(14)–C(15)–C(16)	118·9 (4)	117·5 (8)	117·0 (8)
C(11)–C(16)–C(15)	121·3 (5)	120·2 (8)	120·3 (8)
C(11)–C(16)–C(17)	117·3 (5)	118·7 (8)	119·2 (8)
C(15)–C(16)–C(17)	121·4 (4)	120·9 (9)	120·3 (8)
C(10)–C(17)–C(16)	123·7 (5)	122·8 (9)	123·2 (8)
C(10)–C(17)–C(18)	117·0 (5)	118·2 (9)	117·2 (9)
C(16)–C(17)–C(18)	119·4 (5)	118·9 (10)	119·6 (9)
C(6)–C(18)–C(7)	122·0 (6)	121·5 (10)	120·4 (9)
C(6)–C(18)–C(17)	117·0 (5)	118·3 (10)	118·8 (9)
C(7)–C(18)–C(17)	120·9 (6)	119·8 (9)	120·9 (9)

cinogenicity. Thus, any theory of carcinogenicity must account for both flat and buckled molecules.

A projection of the crystal structure, viewed along **b**, is shown in Fig. 2. The ordered molecules pack along the line $x = \frac{1}{2}$, while the disordered molecules, which lie in the same plane related to each other by a pseudo mirror plane at $z = \frac{1}{4}, \frac{3}{4}$, pack along the line $x = 0$. The dihedral angles and interplanar spacings between like molecules packing along the twofold screw axes are 75·9 (1), 73·7 (2), 69·7 (2)° and 3·418 (7), 3·25 (2) and 3·17 (1) Å, respectively, for the ordered and the two disordered molecules, respectively. Views of the surroundings of a molecule are shown in Fig. 3. The methyl group [C(19)] overlaps C(11) of the 'K region' (a region corresponding to the reactive 9–10 double bond in phenanthrene) of a molecule related by a **b**

Ordered molecule A

Disordered molecule B
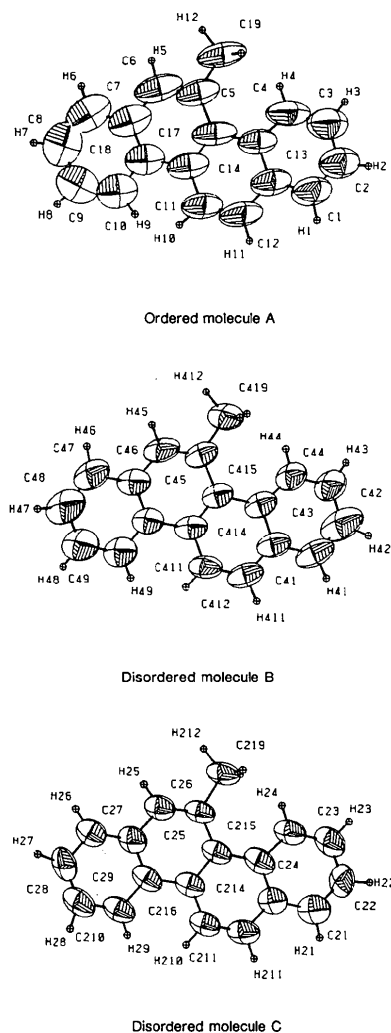
Disordered molecule C

Fig. 1. Molecular skeleton with numbering of non-H atoms and diagrams of 50% probability thermal ellipsoids of the three types of molecule. These diagrams were drawn using the CHEMGRAF program system (Davies, 1983).

around the 'bay region'; C(19) lies 0·20–0·44 Å from the least-squares best plane through C(1) to C(18). The torsion angles around the bay region also are large as seen in Table 3. The values, which range between 2 and 13°, are similar to those in 5,12-dimethylchrysene (Zacharias, Hecht & Loy, 1977), but are less than those observed for 7,12-dimethylbenz[a]anthracene which has torsion angles of 17·8 (4)–21·9 (4)° in the bay region (Iball, 1964; Zacharias & Glusker, unpublished; Glusker, 1981). It appears that steric hindrance in monomethylchrysenes can be relieved by in-plane distortions (such as increases in bond angles) more readily than it can in methylbenz[a]anthracenes with a less distortable carbon skeleton. Our study also indicates that while some carcinogenic polycyclic aromatic hydrocarbons are highly distorted in the 'bay region', such distortions are not required for car-

translation. There are no unusually short intermolecular contacts between any pairs of molecules. We conclude, then, that the disorder has occurred by random packing of molecules B and C in the available space resulting from packing of molecules A. Intermolecular interaction energies for nearest neighbors (Kitaigorodskii, 1973) gave substantially the same values of $-84 \cdot 3$ (1) and $-84 \cdot 5$ (1) kJ mol^{-1} for two hypothetical structures composed of molecules A and B and molecules A and C, respectively. Crystal structures composed of both ordered and disordered molecules in the same structure have been found for *trans*-azobenzene (Brown, 1966)

and *trans*-stilbene (Hoekstra, Meertens & Vos, 1975). The disorder in benzanilide, which contains phenyl rings comparable to rings I and IV of (I), has also been described (Kashino, Ito & Haisa, 1979).

Molecular overlap in 5-methylchrysene A—A—A

(a)

Molecular overlap in 5-methylchrysene B- -C- -B

(b)

Table 3. *Deviations* (Å) *of the atoms from the ring planes and the dihedral angles* (°) *with the planes*

Root-mean-square deviations

	I	II	III	IV
Ordered molecule	0·014 (8)	0·008 (7)	0·013 (7)	0·002 (9)
Disordered molecule B	0·03 (2)	0·03 (1)	0·02 (1)	0·02 (2)
Disordered molecule C	0·02 (2)	0·03 (1)	0·03 (1)	0·01 (2)

Dihedral angles between rings	I II	II III	III IV	I III	II IV
Ordered molecule	2·2 (3)	1·5 (3)	2·1 (3)	3·6 (3)	1·6 (3)
Disordered molecule B	4·3 (7)	5·1 (5)	0·6 (7)	9·2 (6)	4·5 (7)
Disordered molecule C	7·3 (6)	3·9 (5)	3·8 (6)	11·3 (5)	3·5 (6)

Maximum deviations and deviation of C(19) from the plane through rings I, II, III and IV

	Maximum deviations	Deviation of C(19)
Ordered molecule	−0·083 (6) at C(5)	−0·198 (8)
	0·061 (10) at C(3)	
Disordered molecule B	−0·21 (2) at C(3)	0·44 (1)
	0·18 (1) at C(5)	
Disordered molecule C	−0·24 (2) at C(3)	0·44 (1)
	0·24 (1) at C(5)	

Torsion angles (°)	19–5–15–14	5–15–14–4	10–17–16–11
Ordered molecule	−4·4 (8)	−4·5 (8)	2·0 (8)
Disordered molecule B	12 (2)	8 (2)	7 (2)
Disordered molecule C	−4 (1)	−13 (1)	6 (2)

Angles (°) in bay region	19–5–15	5–15–14	15–14–4	19–5–6
Ordered molecule	125·7 (5)	122·5 (4)	127·0 (5)	116·2 (5)
Disordered molecule B	125·6 (7)	123·9 (8)	125·2 (8)	115·7 (8)
Disordered molecule C	125·9 (7)	124·5 (7)	124·4 (8)	115·8 (8)

Molecules in chrysene in planes 3.4 Å apart.

(c)

Fig. 3. Stacking of molecules in layers. The least-squares best plane has been drawn through the ring system with black bonds. Molecules with open bonds lie approximately 3·4 Å from this plane. (a) Ordered molecules $A \cdots A \cdots A$ (also disordered molecules $B \cdots B \cdots B$ and $C \cdots C \cdots C$). (b) Disordered molecules $C \cdots B \cdots C$ (also disordered molecules $B \cdots C \cdots B$). (c) Chrysene (Burns & Iball, 1960; Munnoch & Wright, 1974, 1975).
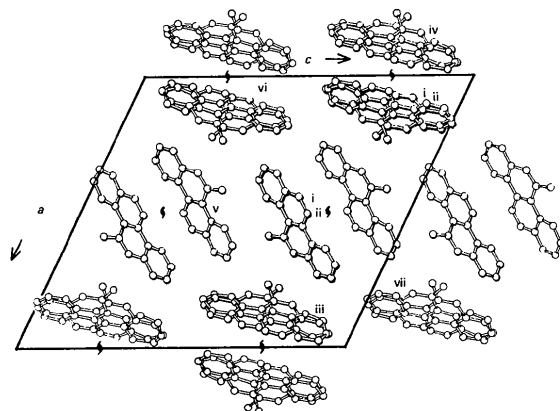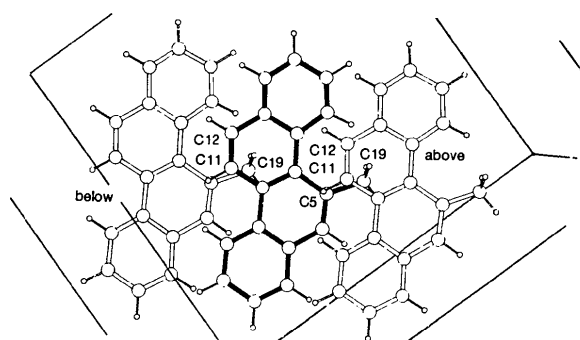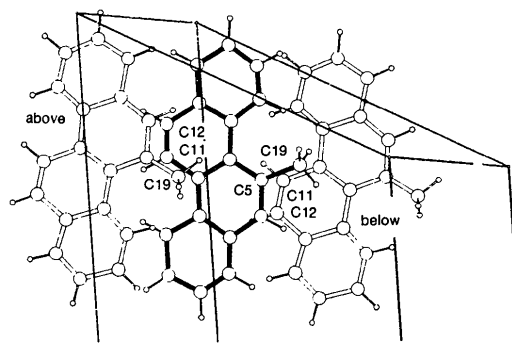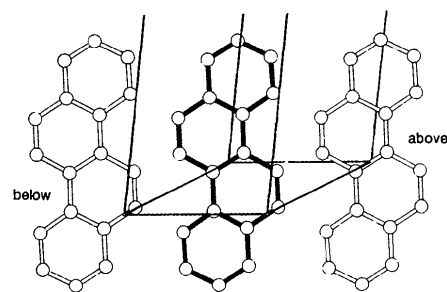
Fig. 2. Projection of the crystal structure viewed along **b**. Molecules B and C are disordered with respect to each other. One translation in each **a** and **c** is shown. Symmetry code: (i) x, y, z; (ii) $x, 1 + y, z$; (iii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iv) $-x, -\frac{1}{2} + y, \frac{3}{2} - z$; (v) $1 - x, -y, 1 - z$; (vi) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (vii) $1 - x, -y, 2 - z$.

The metabolizing enzyme acts on 5-methylchrysene to give a diol epoxide. The carcinogenically active species is 3,4-epoxy-1,2,3,4-tetrahydro-5-methyl-1,2-chrysenediol rather than the 9,10-epoxy-7,8,9,10-tetrahydro-7,8-diol (Melikian, LaVoie, Hecht & Hoffmann, 1982, 1983). This fact must be related to the location of the 5-methyl group which either aids in orienting 5-methylchrysene in the active site of the metabolizing enzyme or else causes more distortion in the resulting diol epoxide, thus enhancing its carcinogenic activity.

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Structure of 1,4,5,8-Tetramethylnaphthalene, C14H16

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Abstract. $M_r = 184.28$, $P2_1/n$, $a = 7.962$ (3), $b = 5.205$ (1), $c = 12.922$ (4) Å, $\beta = 104.98$ (2)°, $V = 517.3$ (2) Å^3, $Z = 2$, $D_x = 1.18 \text{ g cm}^{-3}$, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.62 \text{ cm}^{-1}$, $F(000) = 200$, 300 K, final conventional $R = 0.045$ for 607 unique reflections and 96 parameters. The carbon framework is planar, relieving short intramolecular contacts between the methyl groups by in-plane distortions of the bond angles and bond lengths. The structure is compared with those of related compounds.

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