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Refinement of Di-*para*-anthracene*

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Abstract. $(C_{14}H_{10})_2$, orthorhombic, *Pbca*, a = 12·085 (4), b = 18·853 (7), c = 8·139 (3) Å, Z = 4, D_x = 1·28 Mg m^{-3}; R = 0·040 and R_w = 0·042 for 1019 observed intensities. The crystal structure has been refined by using single-crystal X-ray diffraction data. The molecule consists of two bent anthracenes bridged by a pair of long C—C bonds (1·624 Å), and possesses pseudo *mmm* symmetry.

Introduction. The crystal structure of di-*para*-anthracene has been determined by Ehrenberg (1966). The data used in the study were a limited number of reflection intensities measured by visual comparison of Weissenberg photographs from two different crystals. The present study was intended to supplement Ehrenberg's work by using accurate single-crystal X-ray diffraction data.

A small crystal, about 0·2 mm diameter and 0·3 mm long, was grown from an acetone solution of octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine mixed with anthracene by photo-dimerization under ultraviolet light. Two equivalent sets of reflections within the limiting sphere of 0·65 Å^{-1} in sin θ/λ were measured with Mo $K\alpha$ radiation, with an ω–2θ continuous scan and a scan speed of 1° min^{-1}. The intensities of three standard reflections (600, 010, and 002), measured periodically after every fifty reflection measurements, decreased gradually to 60% of their original intensities at the end. Therefore, the observed data were corrected for the average decrease obtained from these standard reflections. The agreement between the equivalent reflections was satisfactory, with a conventional R = 0·020. 1609 independent reflection data were collected, of which only 1019 had observable intensities according to the criterion $I \geq 3\sigma(I)$, where $\sigma = (I + B)^{1/2}$, I and B being the peak and background intensities respectively. The structure was solved by a direct method using XRAY 76 (Stewart, 1976). H atoms were located by a difference Fourier synthesis.

Table 1. *Positional parameters of di-para-anthracene*

For C atoms B is the equivalent value of the anisotropic temperature factor.

	x	y	z	B (Å^2)
C(1)	0·4062 (2)	0·1285 (1)	0·2313 (3)	4·17
C(2)	0·3104 (2)	0·1669 (1)	0·1945 (3)	4·78
C(3)	0·2363 (2)	0·1404 (1)	0·0812 (3)	4·80
C(4)	0·2554 (2)	0·0755 (1)	0·0064 (3)	4·16
C(5)	0·3903 (2)	−0·1538 (1)	0·1043 (3)	4·25
C(6)	0·4376 (2)	−0·1975 (1)	0·2223 (3)	4·87
C(7)	0·5153 (2)	−0·1710 (1)	0·3302 (3)	4·82
C(8)	0·5455 (2)	−0·0999 (1)	0·3216 (3)	4·17
C(9)	0·5309 (2)	0·0214 (1)	0·1848 (2)	3·53
C(10)	0·3792 (2)	−0·0328 (1)	−0·0390 (3)	3·68
C(11)	0·4222 (2)	−0·0834 (1)	0·0918 (3)	3·53
C(12)	0·4997 (2)	−0·0560 (1)	0·2034 (2)	3·46
C(13)	0·4264 (2)	0·0640 (1)	0·1557 (2)	3·51
C(14)	0·3499 (2)	0·0366 (1)	0·0432 (2)	3·57
H(1)	0·463 (2)	0·150 (1)	0·305 (2)	1·9 (5)
H(2)	0·293 (2)	0·213 (1)	0·248 (3)	2·5 (5)
H(3)	0·167 (2)	0·167 (1)	0·058 (3)	4·1 (7)
H(4)	0·203 (2)	0·056 (1)	−0·071 (3)	2·8 (6)
H(5)	0·334 (2)	−0·171 (1)	0·026 (3)	2·4 (5)
H(6)	0·417 (2)	−0·249 (1)	0·232 (3)	3·2 (6)
H(7)	0·555 (2)	−0·203 (1)	0·415 (3)	3·2 (6)
H(8)	0·606 (2)	−0·080 (1)	0·395 (2)	1·3 (5)
H(9)	0·567 (2)	0·038 (1)	0·290 (2)	0·9 (4)
H(10)	0·314 (2)	−0·053 (1)	−0·090 (2)	0·8 (4)

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* Bi(9,10-dihydro-9,10-anthracenediyl).

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Full-matrix least-squares refinement reduced the R indices to $R = 0{\cdot}040$ and $R_w = 0{\cdot}042$ for the 1019 observed intensities. An isotropic secondary-extinction correction factor (Zachariasen, 1968) was included in the refinement (largest factor, $F_c/F_{\text{corr}} = 0{\cdot}50$, for the 020 reflection). The final least-squares parameters are given in Table 1.* The unit-cell dimensions, determined from nine high-angle reflections, are given in the *Abstract.*

Discussion. This study confirms the crystal structure determined by Ehrenberg (1966), within a maximum discrepancy of 0·06 Å in the bond lengths and 3° in the bond angles. The bond lengths and angles of the C—C bonds are given in Fig. 1. The standard deviations are 0·003 Å and 0·3° for all the C—C bonds. Each anthracene component is bent in the middle by about 48·8° into two virtually identical planar segments. The two anthracene components of the dimer are related by inversion symmetry, and are bridged by a pair of long C—C bonds (1·624 Å) across the apex atoms, as shown in Fig. 1. The non-bonded intramolecular distances, C(11)···C(13′) = 2·827 (3) and C(12)···C(14′) = 2·832 (3) Å, are extremely short compared to the normal separation of aromatic molecules of 3·4 Å. If the four covalent bonds from each apex C atom are arranged in an ideal tetrahedral geometry with bond lengths of 1·54 Å, the bending of the anthracene molecule at the apex should be 44·4° and the two intramolecular distances, C(11)···C(13′) and C(12)···C(14′), should be 2·55 Å, which is extremely short. The packing strain arising from this close approach must be responsible for the elongation of the two bridging bonds [C(9)—C(10′) = 1·624 Å] and also for the excessive bending (48·8°) of the anthracene monomer. The benzene rings at the ends of the anthracene molecules are nearly regular hexagons, with almost identical bond lengths (1·39 ± 0·01 Å) and bond angles (120 ± 1°). The atoms in each ring are coplanar, with maximum deviations from the mean planes of 0·007 Å for the C(1) ring and 0·012 Å for the C(8) ring. The atoms C(2), C(6′), C(3′) and C(7) are also coplanar with a maximum deviation of 0·002 Å, and the atoms C(13), C(11′), C(14′) and C(12) are almost on the plane, with a maximum deviation of 0·02 Å. The mean planes of several other groups of atoms and their angular relations have also been calculated and are presented in Table 2. The three mean planes given in Table 2, of the C(9) group, the C(6′) group and the C(2) group [or the C(11) group], are mutually perpendicular to each other within 0·3°. The dihedral angles made by the C(1) benzene ring and the above three mean planes are almost the same as the corresponding dihedral angles of the C(8) ring, with a maximum difference of 0·8°. Comparison of the mutual orientations of several key atom groups, as given in Table 2, and a related study of the atomic distances from each mean plane, indicate that the molecule possesses pseudo *mmm* symmetry, even though its site has inversion symmetry only. This highly symmetric shape of the molecule is an indication of easy intermolecular packing (without excess strain) which can be seen from the intermolecular distances as discussed below.

The shortest intermolecular distances, which are in the range of van der Waals interactions, occur mostly

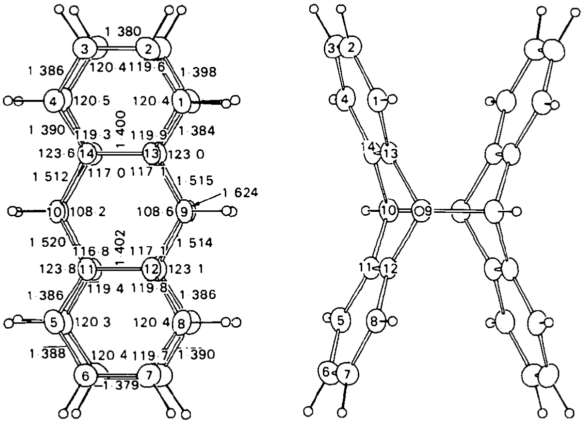

Fig. 1. *ORTEP* drawings (Johnson, 1965) of the di-*para*-anthracene molecule viewed from two different directions, showing the numbering of the C atoms. Equivalent atoms related by the center of symmetry are indicated with primed numbers throughout the text.

Table 2. *Mean planes of several groups of atoms and their dihedral angles*

The largest deviations of atoms from the corresponding mean planes are 0·007 Å for the C(1) ring, 0·012 Å for the C(8) ring, 0·003 Å for the C(11) atom group, 0·015 Å for the C(2) atom group, and 0·0002 Å for the C(6′) atom group. X, Y, and Z are in Å.

C(1) benzene ring	$0{\cdot}4868X + 0{\cdot}4656Y - 0{\cdot}7391Z - 2{\cdot}1268 = 0$
C(8) benzene ring	$0{\cdot}7329X - 0{\cdot}2444Y - 0{\cdot}6350Z - 3{\cdot}6365 = 0$
C(9), C(10), C(9′), C(10′)	$-0{\cdot}3562X + 0{\cdot}9209Y - 0{\cdot}1582Z + 2{\cdot}1526 = 0$
C(11), C(12), C(13), C(14)	$0{\cdot}6576X + 0{\cdot}1270Y - 0{\cdot}7462Z - 2{\cdot}6042 = 0$
C(2), C(3), C(6), C(7)	$0{\cdot}6599X + 0{\cdot}1232Y - 0{\cdot}7412Z - 1{\cdot}7048 = 0$
C(2), C(6′), C(3′), C(7)	$-0{\cdot}6593X - 0{\cdot}3698Y - 0{\cdot}6547Z + 4{\cdot}6731 = 0$

Dihedral angles (°)

Atom group	C(1) ring*	C(8) ring*	C(9) group	C(11) group	C(2) group
C(8) ring*	44·6 (3)				
C(9) group*	−68·1 (2)	67·3 (3)			
C(11) group*	21·9 (4)	−22·7 (4)	90·0 (3)		
C(2) group*	22·1 (2)	−22·4 (2)	90·2 (2)	0·0 (4)	
C(6′) group	90·5 (2)	88·7 (2)	90·1 (2)	89·7 (4)	89·7 (1)

* The sign of the dihedral angle indicates, by analogy with torsional angles, the rotation direction of the plane in the first column about the C(10) → C(9) vector in a right-handed system.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35353 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

between H atoms [*i.e.* H(3)···H(6) = 2·43 (3), H(1)···H(6) = 2·41 (3), and H(1)···H(7) = 2·49 (3) Å] and also between H and C atoms [*i.e.* C(7)···H(3) = 2·87 (3) and C(4)···H(9) = 2·91 (2) Å]. All the C···C intermolecular distances are longer than 3·7 Å.

Table 3. *Rigid-body analysis of the thermal motion of the di-para-anthracene molecule*

T (Å²) and **L** (deg²) relative to **a**, **b** and **c**

$$\mathbf{T} = \begin{pmatrix} 0{\cdot}0379\,(7) & -0{\cdot}0077\,(6) & -0{\cdot}0023\,(7) \\ & 0{\cdot}0500\,(6) & -0{\cdot}0044\,(6) \\ & & 0{\cdot}0381\,(8) \end{pmatrix}$$

$$\mathbf{L} = \begin{pmatrix} 3{\cdot}9\,(3) & 0{\cdot}0001\,(1) & -0{\cdot}0009\,(1) \\ & 3{\cdot}9\,(6) & 0{\cdot}0001\,(1) \\ & & 7{\cdot}4\,(3) \end{pmatrix}$$

Principal axes of libration

	R.m.s. amplitude (°)	Direction cosines		
L_1	3·02	0·499	−0·035	−0·866
L_2	1·97	−0·170	−0·984	−0·058
L_3	1·45	−0·849	0·176	−0·497

The rigid-body motions of the di-*para*-anthracene molecule were determined from the thermal-ellipsoid data of all the C atoms by the method of Schomaker & Trueblood (1968). The results are given in Table 3. The librations are considerably anisotropic, with the largest principal axis lying almost on the C(9), C(10), C(9′), C(10′) plane. These librational motions give rise to increases in the C—C bond lengths in the range of 0·0016 [for C(9)—C(10)] to 0·0028 Å [for C(9)—C(13)], which are not greater than one standard deviation of the measured bond length.

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Structure de la Phénoxy-3 Thiolanone-2

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Abstract. $C_{10}H_{10}O_2S$, monoclinic, $P2_1/n$, a = 11·572 (3), b = 10·353 (3), c = 8·260 (2) Å, γ = 108·50 (20)°, Z = 4. The structure was solved by direct methods. Full-matrix least-squares refinement converged at R = 0·096 and R_w = 0·064 for all the 1514 observed reflexions. The five-membered thiolanone ring exhibits a structure very close to an envelope conformation. The C(2) substituent is located in an equatorial position.

Introduction. L'interprétation des données spectroscopiques relatives aux dérivés de la γ-thiobutyrolactone (Lere-Porte, Galsomias & Petrissans, 1980) nous a conduit à déterminer la structure de la phénoxy-3 thiolanone-2 par diffraction des rayons X. A notre connaissance, il existe peu d'études structurales consacrées au cycle thiolanone. Les déterminations effectuées antérieurement (Shefter, 1968; Freer & Kraut, 1965) ont permis néanmoins de montrer que la thiolphtalide est une molécule plane tandis que le chlorhydrate d'homocystéine présente une conformation de type enveloppe, le groupe NH_3^+ occupant une position équatoriale. Les mesures d'intensité ont été effectuées au diffractomètre Philips PW 1100 en utilisant la radiation Cu $K\alpha$. 1514 réflexions ont été mesurées. Les positions des atomes de C,O,S ont été déterminées à l'aide du programme *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977), celles des atomes H par série différence. Deux atomes H appartenant au cycle lactonique et au noyau

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