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The Molecular Structure of Bi(anthracene-9,10-dimethylene), $C_{32}H_{24}$

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(*Received* 31 *March* 1976; *accepted* 28 *June* 1976)

The structure of bi(anthracene-9,10-dimethylene) (β form) was determined by single-crystal X-ray diffraction techniques. The compound crystallizes in the monoclinic system, space group $P2_1/c$ with two molecules per unit cell. Unit-cell parameters are: $a = 10{\cdot}277$ (1), $b = 12{\cdot}794$ (2), $c = 8{\cdot}466$ (1) Å, $\beta = 112{\cdot}89$ (1)°. The symmetry of the molecule was examined by applying a least-squares method and a statistical test. It was found that the molecule in the crystal shows significant deformation from the ideal models, which have symmetries D_{2h} or C_{2h}.

Introduction

Golden (1961) reported two crystalline modifications of bi(anthracene-9,10-dimethylene), α and β forms, and gave the X-ray single-crystal data of these and of the photoisomer. Milledge (1962) reported a preliminary X-ray analysis of the α and β forms, and Ehrenberg (1966*a,b*) performed the structure analysis on the photoisomer and found that it has highly strained four-membered rings with anomalous long C–C single bonds. She also gave brief structural information on the β form. However, the molecular structure has not yet been described.

Recently (Ferguson, Mau & Morris, 1973; Ferguson & Mau, 1974) the spectral and photochemical properties of various sandwiched anthracene dimers have been studied and it has been found that the photoisomerization of bi(anthracene-9,10-dimethylene) occurs even at 4 K. This means that the molecule has a favourable conformation for photoisomerization to occur. Precise structural information on this molecule is desired for further investigation of the mechanism of the inter-ring interaction.

In this paper an X-ray analysis of the β form of the title compound is presented.

Experimental

Preliminary X-ray photographs showed that the crystals are monoclinic with space group $P2_1/c$. The space group was chosen as $P2_1/a$ in earlier investigations, but we have altered the axis in accordance with the usual convention. The unit-cell parameters were refined by a

Table 1. *Crystal data*

Bi(anthracene-9,10-dimethylene), $C_{32}H_{24}$
$M_r = 408{\cdot}55$
Monoclinic
$a = 10{\cdot}277 \pm 0{,}001^*$ Å
$b = 12{\cdot}794 \pm 0{\cdot}002$
$c = 8{\cdot}466 \pm 0{\cdot}001$
$\beta = 112{\cdot}89 \pm 0{\cdot}01°$

$V = 1025{\cdot}4$ Å^3
$D_x = 1{\cdot}322$ g cm^{-3}
$Z = 2$
$F(000) = 432$
Space group $P2_1/c$
$\mu = 6{\cdot}85$ cm^{-1} (Cu $K\alpha$)
λ(Cu $K\alpha$) = $1{\cdot}54178$ Å

* The errors given are twice the estimated standard deviations and are significant at the 5% level.

least-squares fit to 12 independent reflexion angles on a Hilger & Watts four-circle diffractometer. Crystal data are listed in Table 1.

A crystal with dimensions 0·13 × 0·16 × 0·35 mm was mounted for the data collection. The intensity measurements were performed on the diffractometer with the θ–ω step scan technique ($\theta < 72°$, 0·02° per step, counting 2 s per step and 50 steps per reflexion) and Ni-filtered Cu $K\alpha$ radiation. The intensities of 1968 reflexions were measured; 1865 were non-zero and were used in the refinement.

Table 2. *Final atomic fractional coordinates* ($\times 10^5$ *for* C *atoms and* $\times 10^4$ *for* H *atoms*), *thermal parameters* ($\times 10^4$ *for anisotropic parameters and* $\times 1$ *for isotropic*) *and their estimated standard deviations*

The temperature factors are expressed in the forms: $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{23}kl + B_{31}lh)]$ or $\exp[-B(\text{iso})(\sin\theta/\lambda)^2]$. The isotropic thermal parameters of the C atoms were estimated by approximating the thermal ellipsoids to spheres of the same volume.

	x	y	z	B(iso)
C(1)	73062 (22)	21471 (15)	6750 (28)	4·23
C(2)	86946 (22)	21052 (17)	16845 (32)	5·21
C(3)	92099 (22)	13639 (18)	29941 (31)	5·33
C(4)	83230 (22)	6715 (16)	32652 (28)	4·40
C(5)	34813 (24)	−2086 (18)	25488 (29)	4·77
C(6)	20977 (25)	716 (20)	19207 (34)	5·60
C(7)	15685 (23)	8106 (19)	5964 (33)	5·41
C(8)	24395 (22)	12640 (16)	−787 (29)	4·49
C(9)	48414 (21)	15111 (14)	−525 (26)	3·26
C(10)	58936 (20)	79 (15)	25854 (24)	3·30
C(11)	62959 (20)	14694 (14)	9468 (25)	3·22
C(12)	68251 (21)	7046 (14)	22892 (24)	3·24
C(13)	44484 (20)	2776 (14)	19471 (25)	3·37
C(14)	39149 (20)	10365 (14)	5911 (25)	3·33
C(15)	42747 (22)	19454 (15)	−18697 (27)	4·05
C(16)	63412 (23)	−10853 (16)	32799 (29)	4·23
H(1)	6982 (17)	2636 (15)	−227 (24)	2·8 (5)
H(2)	9390 (18)	2671 (16)	1467 (27)	4·9 (6)
H(3)	10281 (20)	1369 (15)	3753 (27)	4·8 (6)
H(4)	8691 (18)	132 (14)	4168 (25)	3·5 (5)
H(5)	3836 (17)	−764 (14)	3471 (24)	3·2 (5)
H(6)	1475 (18)	−255 (15)	2439 (26)	4·3 (6)
H(7)	587 (18)	1058 (15)	189 (26)	3·7 (6)
H(8)	2043 (18)	1806 (14)	−1031 (25)	3·2 (5)
H(15*a*)	5104 (19)	2297 (15)	−2166 (27)	4·4 (6)
H(15*b*)	3567 (16)	2502 (16)	−2025 (24)	3·5 (5)
H(16*a*)	7511 (19)	−1167 (15)	3734 (26)	4·2 (6)
H(16*b*)	6179 (18)	−1273 (15)	4216 (25)	3·9 (6)

Table 2 (*cont.*)

	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{31}
C(1)	1581 (35)	527 (16)	1744 (49)	−157 (37)	23 (46)	1559 (70)
C(2)	1310 (34)	856 (21)	2452 (59)	−377 (43)	−413 (59)	1625 (77)
C(3)	1187 (34)	879 (20)	2360 (60)	−66 (43)	−414 (60)	858 (75)
C(4)	1237 (32)	649 (17)	1664 (47)	157 (38)	−192 (49)	528 (66)
C(5)	1795 (40)	796 (19)	1711 (50)	−770 (45)	−565 (51)	1778 (76)
C(6)	1648 (39)	1190 (24)	2538 (62)	−1207 (53)	−1296 (66)	2383 (85)
C(7)	1121 (33)	1076 (23)	2697 (65)	−361 (46)	−1470 (66)	1155 (78)
C(8)	1174 (32)	710 (18)	1948 (52)	−9 (39)	−830 (52)	640 (68)
C(9)	1373 (31)	345 (12)	1294 (39)	154 (33)	1 (40)	1023 (59)
C(10)	1403 (31)	487 (13)	862 (35)	4 (35)	−105 (40)	642 (56)
C(11)	1196 (30)	422 (13)	1237 (39)	97 (33)	−186 (40)	1020 (58)
C(12)	1237 (29)	430 (13)	1133 (38)	110 (33)	−225 (39)	856 (57)
C(13)	1344 (32)	492 (14)	1153 (40)	−300 (35)	−381 (40)	1111 (59)
C(14)	1159 (29)	457 (13)	1289 (41)	55 (34)	−470 (40)	764 (58)
C(15)	1584 (35)	499 (14)	1383 (44)	257 (38)	306 (44)	703 (65)
C(16)	1868 (38)	532 (15)	1213 (42)	−39 (41)	352 (44)	436 (67)

The structure amplitudes were obtained by the usual data reduction procedure. The estimated standard deviation in the intensity of each reflexion was taken to be $\sigma(I) = [\sigma_{\text{c.s.}} + 1{\cdot}398 \times 10^{-5} I^2]^{1/2}$, where $\sigma_{\text{c.s.}}$ is the estimated standard deviation of the intensity from the counting statistics and the coefficient of I^2 was derived from the deviations of the intensities of the reference reflexions.

Determination and refinement of the structure

The phases of the normalized structure factors were determined by symbolic addition (Karle & Karle, 1966). The first E map revealed the complete structure except for the H atoms. All the H atoms were located in a difference Fourier map calculated after four cycles of refinement with anisotropic thermal factors for the C atoms. The final refinement was performed by minimizing the function $\Sigma\{|F_o - F_c|^2/\sigma(F_o)^2\}$ with isotropic thermal factors for the H atoms. The discrepancy indices at convergence were as follows: $\Sigma\,|F_o - F_c|/\Sigma\,|F_o| = 0{\cdot}069$,

$$\frac{\Sigma\{|F_o - F_c|^2/\sigma(F_o)^2\}^{1/2}}{\Sigma\{|F_o|^2/\sigma(F_o)^2\}} = 0{\cdot}040.$$

Final atomic parameters are listed in Table 2 with their estimated standard deviations.*

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* A table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32035 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1968).

Discussion

Bond lengths and angles are shown in Fig. 1. All the values in the anthracene component are normal; in particular, C(6)–C(7), whose equivalent dimensions in di-*p*-anthracene and the photoisomer were reported to be short (Ehrenberg, 1966*a,b*), is 1·404 Å. The bond length C(15)–C(16)′, 1·565 Å, is a little longer than the normal value of 1·54 Å, which may be due to the repulsive force between the two half molecules. However, it is not as long as the corresponding bond in di-*p*-xylylene, 1·63 Å, reported by Lonsdale, Milledge & Rao (1960).

The side view of the molecule is shown in Fig. 2 with the interatomic distances between the anthracene rings. The distance C(9)· · · C(10)′, 2·772 Å, is slightly longer than the value, 2·70 Å, estimated by Milledge (1962), but it is much longer (by ~1·0 Å) than that of the photoisomer (1·77 Å). This distance is apparently shorter than the normal van der Waals contact, and a high quantum yield of the photo-chemical reaction, which connects the facing atoms, C(9) and C(10)′, and C(10) and C(9)′, is reasonably explained by this geometry.

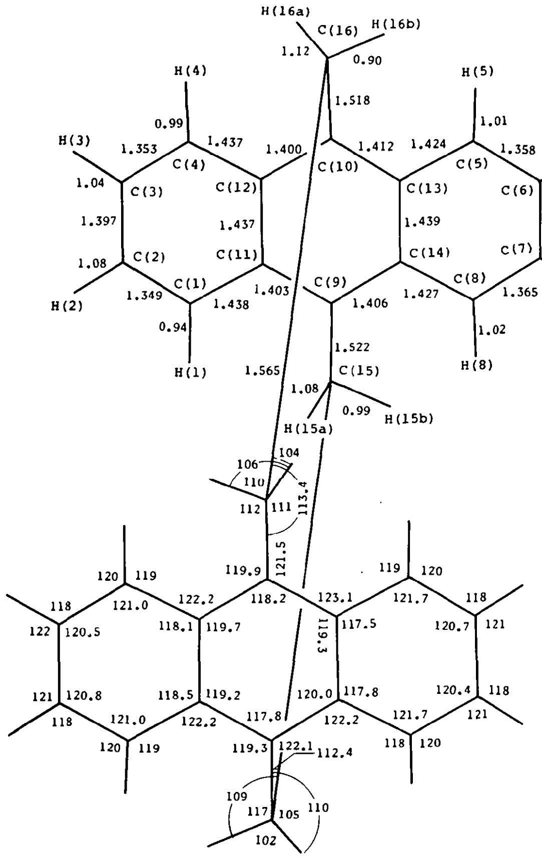

Fig. 1. Bond lengths (Å) and angles (°). The average e.s.d.'s of the bond lengths and angles are 0·003, 0·02 Å, 0·2, 1, and 2° for C–C, C–H, C–C–C, C–C–H and H–C–H respectively.

Fig. 3 gives the deviations of the atoms from the following least-squares planes: plane 1: $-0.24695x + 0.69175y + 0.67860z' - 0.30617 = 0$, plane 2:

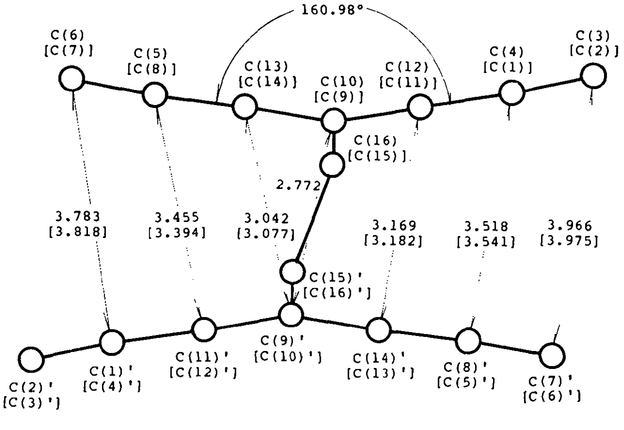

Fig. 2. Side view of the molecule. Interatomic distances (Å) between the anthracene components are also given. The average e.s.d. of the distances is 0·003 Å.

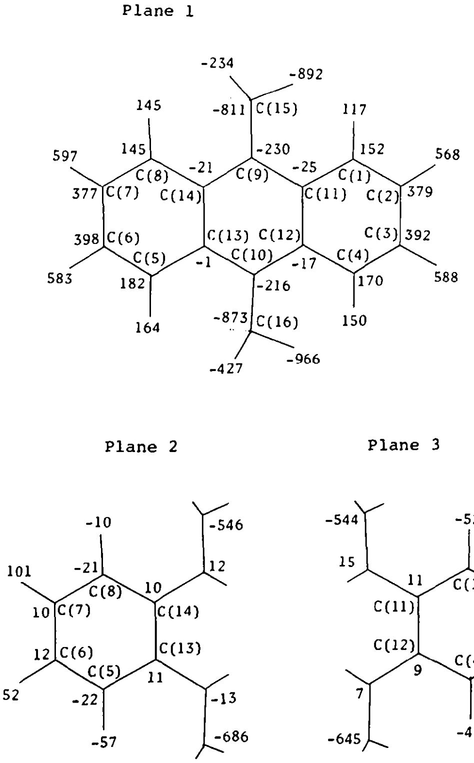

Fig. 3. Deviations (×10³ Å) of atoms from the least-squares planes. Each plane is defined by the atoms indicated.

$-0.08624x+0.72941y+0.67862z'-0.94004=0$, plane 3: $-0.40489x+0.64873y+0.64436z'+0.80856=0$, where x, y, and z' are coordinates in Å along **a**, **b**, and **c***.

The deviations show that the anthracene component is bent around the central C atoms; the bending angle was estimated to be 19·0° from the normal vectors of the side benzene rings, and this value is about half that found in the photoisomer (Ehrenberg, 1966*b*), C(1), C(4), C(5) and C(8) lie below the least-squares planes determined by the side benzene rings, and both benzene rings are slightly bent at these atoms.

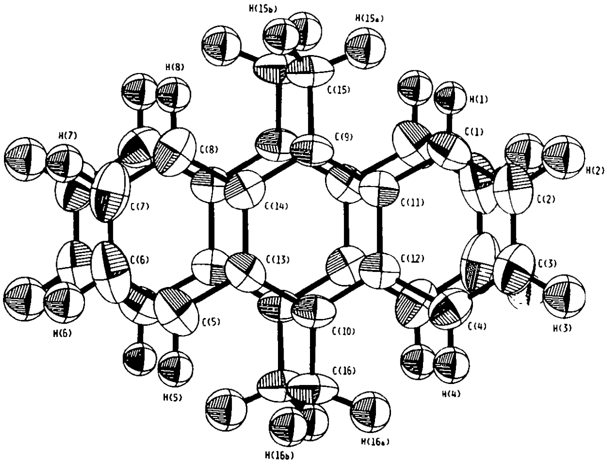

Fig. 4. Molecular framework projected onto the least-squares plane of the anthracene component. Thermal ellipsoids were drawn by *ORTEP* (Johnson, 1965) and scaled to enclose 50% probability.

Fig. 4 shows the molecular structure with thermal ellipsoids projected onto the least-squares plane determined by the anthracene component. The anthracene groups are displaced by about 0·6 Å along their long axes and avoid interfacial contact. Because of this conformation, C(15)–C(16)′ is twisted and the torsion angle C(10)′–C(16)′–C(15)–C(9) is 16·0°. This conformation reduces the steric repulsion between the eclipsed H atoms, H(15*a*) and H(16*b*)′, and H(15*b*) and H(16*a*)′, to some extent.

The molecule appears to have a large vibrational component in the anthracene ring, as can be seen from Fig. 4. A rigid-body motion analysis of the vibration of the C atoms was performed and the results are listed in Table 3. The principal axis of the largest libration component, l_1, makes an angle of 64° with the least-squares plane of the anthracene ring, and is very close to the normal vector of the plane containing C(6), C(7), C(6)′ and C(7)′. Other components are not so noticeable.

As regards the symmetry of the molecule in the crystal, it can be seen from Fig. 4 that it does not have the point group symmetry D_{2h}, but instead has symmetry C_{2h}. Therefore, the molecular symmetry is examined by comparing the ideal structure, having the assumed symmetry C_{2h}, with the real one.

The method of modelling the ideal structure is given below. First of all, we must determine the unitary matrix Z which transforms the atomic coordinates from an arbitrary Cartesian system to one whose axes coin-

Table 3. *Rigid-body motion analysis*

The root-mean-square displacements (r.m.s.d.) are given in degrees (libration) and Å (translation). The unit vectors along the principal axes are referred to the real base vectors **a**, **b**, and **c**.

Moment of inertia

Axis	Moment (at. wt Å^2)	*l*	*m*	*n*
p_1	1729	0·09826	0·02671	0·03057
p_2	2782	0·00898	−0·04344	−0·09344
p_3	3007	−0·03771	0·05923	−0·08232

Libration

Axis	r.m.s.d.	*l*	*m*	*n*
l_1	4·45	−0·05321	−0·05536	−0·08355
l_2	2·24	0·08409	−0·04712	0·04592
l_3	1·70	0·03543	0·02871	−0·08575

Translation

Axis	r.m.s.d.	*l*	*m*	*n*
t_1	0·226	0·10082	0·00987	0·01570
t_2	0·189	−0·00869	0·07677	0·01582
t_3	0·175	−0·03028	0·01084	−0·12627

$$\langle \Delta U^2 \rangle = 0.0048\ \text{Å}^2.$$

Table 4. *Best* Z *matrix and ideal model*

The Z matrix is referred to the orthonormal bases along the *a*, *b* and *c** axes. *x*, *y*, and *z* are the atomic coordinates (×10^5 Å) of the best model satisfying the ideal symmetry C_{2h}, and Δx, Δy, and Δz are the differences (×10^5 Å) between the real and ideal structures.

$$Z=\begin{pmatrix} 0.98756 & 0.15241 & 0.03877 \\ -0.13482 & 0.69352 & 0.70771 \\ 0.08098 & -0.70413 & 0.70544 \end{pmatrix}$$

	x	Δx	y	Δy	z	Δz
C(1)	25502	99	20354	−472	−13850	−40
C(2)	36592	39	23865	−259	−6981	−91
C(3)	36592	−39	23865	259	6981	−91
C(4)	25502	−99	20354	472	13850	−40
C(5)	−23313	−25	14873	579	13944	14
C(6)	−34992	12	15807	303	7015	−21
C(7)	−34992	−12	15807	−303	−7015	−21
C(8)	−23313	25	14873	−579	−13944	14
C(9)	1475	17	13782	−468	−14114	94
C(10)	1475	−17	13782	468	14114	94
C(11)	13184	40	17132	−243	−7179	−23
C(12)	13184	−40	17132	243	7179	−23
C(13)	−10767	−33	14532	307	7190	44
C(14)	−10767	33	14532	−307	−7190	44
C(15)	1884	64	7594	−477	−27990	73
C(16)	1884	−64	7594	477	27990	73

cide with the symmetry axes. The least-squares procedure gives the best Z that minimizes the residual function

$$\sum_{jp}^{NP}|(S^p)^{-1}\,Zx_j^p-\frac{1}{P}\sum_q^P(S^q)^{-1}Zx_j^q|^2.$$

Here S_p and x_j^p denote the matrix of the pth symmetry operation and the positional vector of the jth atom related by the pth symmetry; P is the number of symmetry operations and N is the number of sets of equivalent atoms. The atomic coordinates of the ideal model can be obtained as

$$\frac{1}{P}\,S^p\sum_q^P(S^q)^{-1}\,Z(\text{best})\,x_j^q$$

by averaging the positions of the equivalent atoms transformed by the best Z.

Table 4 shows the atomic coordinates of an ideal model satisfying C_{2h} symmetry together with the deviations from the real one. The deviations along the y axis are obviously systematic and show that the anthracene components are displaced from each other by about 0·04 Å along the short axis. This value seems highly significant, although it may have little influence on the electronic properties of the molecule. It is supposed that the errors in atomic coordinates in the Cartesian system are a sample from a normal distribution with parameters 0, $\sigma^2=(0{\cdot}00285\ \text{Å})^2$, where σ is represented by the maximum value in the estimated standard deviations of the atomic coordinates of the C atoms given in Table 2. The χ^2 test (Snedecor & Cochran, 1967) rejects the null hypothesis that the molecule satisfies the C_{2h} symmetry with probability 99·9% since the calculated χ^2 value, 6700, exceeds 234·0 which is significant at the 0·1% level. Here χ^2 is defined by

$$\sum_j^N\sum_p^P|Z(\text{best})\,x_j^p-\frac{1}{P}S\,\sum_q^P(S^q)^{-1}\,Z(\text{best})\,x_j^q|^2,$$

where the distribution is a χ^2 one with $3N(P-1)$ degrees of freedom.

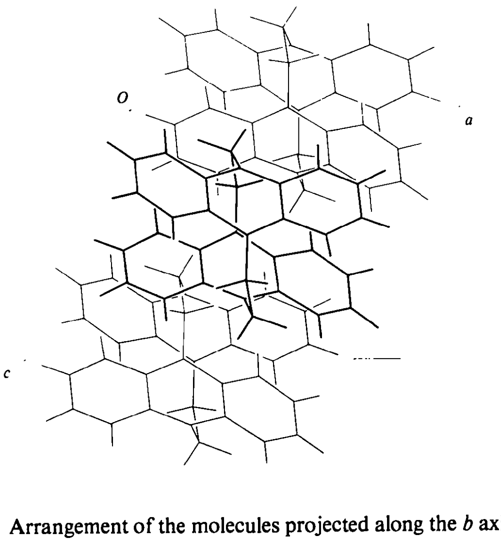

Fig. 5. Arrangement of the molecules projected along the *b* axis.

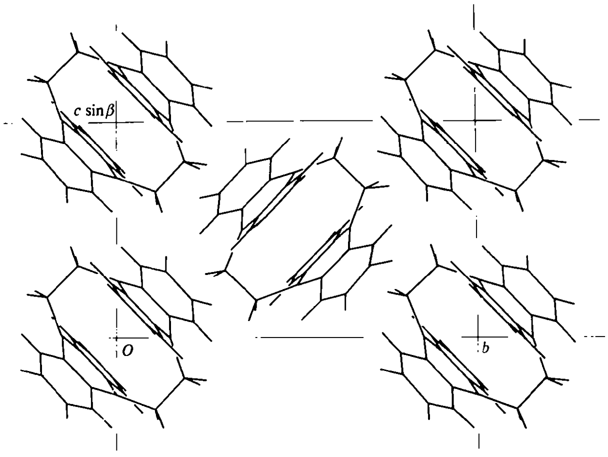

Fig. 6. Arrangement of the molecules projected along the *a* axis.

The best Z matrix gives the directions of the molecular symmetry axes, whose bases are **L**, **M** and **N**;

$$\mathbf{L}=0{\cdot}03686a-0{\cdot}05504b+0{\cdot}09045c,$$
$$\mathbf{M}=0{\cdot}09769a+0{\cdot}01191b+0{\cdot}00497c,$$
and $$\mathbf{N}=0{\cdot}01596a+0{\cdot}05421b+0{\cdot}09074c.$$

Here **L** and **M** are the unit vectors along the short and long axes of the anthracene component, respectively, and **N** is the unit vector which is nearly perpendicular to the anthracene plane. **M** makes an angle of 9·0° with the *a* axis, and **N** 15·2° with the normal vector of (011). This orientation of the molecule can be seen in Figs. 5 and 6, which show the crystal structure projected along the **b** and **a** axes.

Computing programs used in this study were devised in our laboratory by C. Katayama and A. Wada. *RSDA*-4 and *RSMV*-4 of a local version of *UNICS* (Sakurai, 1967) were also used to calculate the bond lengths and angles, and rigid-body motion. All calculations were performed on a FACOM 230-60 computer of Nagoya University Computing Center.

The authors are grateful to Professor S. Misumi of Osaka University for the gift of the sample.

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Acta Cryst. (1977). B33, 360–365

The Crystal Structure of 6b,10b-Dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene, $C_{18}H_{12}$

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(*Received* 7 *June* 1976; *accepted* 9 *July* 1976)

6b,10b-Dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene is monoclinic, space group *Pm*, with $a = 10{\cdot}202\,(5)$, $b = 13{\cdot}849\,(5)$, $c = 8{\cdot}768\,(3)$ Å, $\beta = 103{\cdot}41\,(3)°$. There are four independent molecules in the cell. Least-squares refinement with 1519 reflexions $[I > 3\sigma(I)]$ gave $R = 0{\cdot}032$ for 458 parameters. The molecules are bent about the long bond, 1·606 (4) Å, of the cyclobutane ring. The angles of this highly strained molecule are compared with calculated values, and strain in 1,2-disubstituted benzenes is discussed.

Introduction

The structure of 6b,10b-dihydrobenzo[*j*]cyclobut[*a*]acenaphthylene (II) has been determined as part of a study of the pleiadenes and their precursors (Kolc & Michl, 1973). Other structures in this series which have been determined are 6b,8a-dihydrocyclobut[*a*]acenaphthylene (I) (Hazell, 1976*a*) and 6b,12b-dihydronaphtho[2,3-*j*]cyclobut[*a*]acenaphthylene (III) (Hazell & Weigelt, 1976).

Crystal data

$C_{18}H_{12}$, $M_r = 228{\cdot}3$, monoclinic, $a = 10{\cdot}202\,(5)$, $b = 13{\cdot}849\,(5)$, $c = 8{\cdot}768\,(3)$ Å, $\beta = 103{\cdot}41\,(3)°$; $U = 1205{\cdot}0$ Å^3, $Z = 4$, $D_c = 1{\cdot}26$ g cm^{-3}; space group *Pm*; $\mu(\text{Mo } K\alpha) = 0{\cdot}66$ cm^{-1}. The crystals are prismatic, elongated along [001] and bounded by (010) and (100).

Experimental

The crystals were kindly provided by Josef Michl. A crystal $0{\cdot}60 \times 0{\cdot}35 \times 0{\cdot}25$ mm was mounted along **a**, and intensities were measured out to $2\theta_{\text{max.}} = 50°$ with a Picker FACS-1 diffractometer. Monochromatic Mo $K\alpha$ radiation was used with a scintillation counter and a pulse-height analyser. 1519 independent reflexions with $I > 3\sigma(I)$, according to counting statistics, were used in the subsequent calculations. No correction was applied for absorption.

Determination and refinement of the structure

Since no $0k0$ reflexions with k odd were observed on precession photographs, the space group was assumed to be $P2_1/m$. Direct methods (*MULTAN*, Germain, Main & Woolfson, 1971) showed one well defined

Table 1. *Constrained refinements*

The possibilities considered were (*a*) whether the four molecules are identical and (*b*) could the thermal vibration be best accounted for by refining **T**, **L** and **S** or by refining individual atomic temperature factors. A scale factor and an extinction coefficient were also refined. N_o is the number of observations, N_p the number of parameters.

Coordinates	Thermal parameters	N_o	N_p	R	R_w
(i) Free	U_{ij}	1519	410	0·032	0·037
(ii) Free	**TLS**	1519	240	0·042	0·050
(iii) Identical molecules	U_{ij}	1519	289	0·056	0·069
(iv) Identical molecules	**TLS**	1519	103	0·065	0·085