

## X-ray Structural Analysis of 5,6-Dihydrodibenz[*a,h*]anthracene\*

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The molecular and crystal structure of 5,6-dihydrodibenz[*a,h*]anthracene, C<sub>22</sub>H<sub>16</sub>, has been determined from an X-ray analysis. The compound crystallizes with four molecules in an orthorhombic unit cell of symmetry *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> and dimensions *a* = 8·465 (3), *b* = 15·082 (4), and *c* = 11·616 (3) Å at 22°C. The structure was solved by the application of the symbolic-addition phase determination procedure and refined by the full-matrix least-squares method to *R*(*F*) = 7·5%, based on 854 observed diffractometer data. The solution of the structure was based on the assumption that disorder exists, whereby two molecular sites of partial occupancy, each centered near the origin of the unit cell, are related to each other by a pseudocenter of symmetry. Because of the disorder, the end aromatic rings (I) and (V) both appear to be twisted from the central ring (III) by 10·1 and 6·7° respectively, whereas the expected twist for the end ring adjacent to the ring containing the saturated carbon atoms is about 21°, and that expected for the ring at the opposite (aromatic) end is nearly zero.

### Introduction

In a systematic investigation of the tumorigenic activity of several pentacyclic aromatic hydrocarbons on Swiss mice, it was shown that the compounds have widely varying effects (Lijinsky & Saffiotti, 1965; Lijinsky, Garcia & Saffiotti, 1970). Among the agents tested, 5,6-dihydrodibenz[*a,h*]anthracene was found to be weakly carcinogenic when applied to the skin and apparently not carcinogenic when injected subcutaneously. These observations are in contrast with the potent carcinogenicity of the compound's parent unhydrogenated hydrocarbon, dibenz[*a,h*]anthracene (Lijinsky *et al.*, 1970).

Our interest in obtaining molecular parameters for 5,6-dihydrodibenz[*a,h*]anthracene is an outgrowth of the X-ray study, recently carried out in this laboratory, of another partially hydrogenated polycyclic aromatic hydrocarbon, 5,6-dihydrodibenz[*a,j*]anthracene (Wei, 1972), a potent carcinogen in the skin painting test (Lijinsky *et al.*, 1970). This investigation also affords an opportunity to compare the present crystal structure with that of the parent compound, dibenz[*a,h*]anthracene, which was determined by Robertson & White (1947, 1956) using two-dimensional X-ray analysis.

### Experimental

#### Crystal specimen

The sample of 5,6-dihydrodibenz[*a,h*]anthracene, kindly made available to us by Dr W. Lijinsky of The Eppley Institute for Research in Cancer (now at Oak Ridge National Laboratory), was recrystallized from *n*-hexane solutions. A colorless crystal of triangular pris-

matic habit with length 0·19 mm and sides of the base triangle of 0·09, 0·11, and 0·17 mm, was selected for collecting data. The prism axis of the crystal corresponds to the *a* axis, while the longest base-side nearly coincides with the *c* direction of the unit cell. The crystal was mounted on a glass fiber with the *a* axis nearly parallel to the glass fiber axis.

#### Unit cell and space group

The crystal system and approximate cell parameters were determined from preliminary Weissenberg and precession photographs. The diffractometer coordinates of 10 strong reflections in the 2θ range 64 to 93° were measured on an Oak Ridge computer-controlled diffractometer (Busing, Ellison, Levy, King & Roseberry, 1968) using Cu Kα<sub>1</sub> (λ = 1·54051 Å) radiation, and the cell parameters together with the orientation parameters of the crystal were refined by the method of least squares. The density was determined by flotation in mixtures of 1,2-dichloroethane and *p*-dioxane.

Crystals of C<sub>22</sub>H<sub>16</sub> are orthorhombic with cell parameters and estimated standard errors as follows: *a* = 8·465 (3), *b* = 15·082 (4), and *c* = 11·616 (3) Å. The volume of the unit cell is 1483·0 Å<sup>3</sup>. The observed density of 1·23 (2) g·cm<sup>-3</sup> is close to the calculated value of 1·258 g·cm<sup>-3</sup>, based on four formula units per cell. The total number of electrons per unit cell, *F*(000), is 592. Systematic absences of *h*00 for *h* odd, 0*k*0 for *k* odd, and 00*l* for *l* odd indicate that the space group is probably *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> (*D*<sub>2</sub><sup>4</sup>, No. 19) (see *International Tables for X-ray Crystallography*, 1965, p. 105). This choice was later confirmed by the successful refinement of the structure.

#### X-ray intensity data

A set of three-dimensional data up to 2θ = 105° was recorded with the automatic diffractometer, using Cu Kα radiation. Because the maximum crystal mosa-

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icity in any direction was estimated to be  $2^\circ$ ,\* intensities were measured by an  $\omega$  step-scan technique (H. A. Levy, unpublished work; Brown, 1969). A reference reflection was recorded after every 20 reflections; its intensity fluctuated no more than  $\pm 1.5\%$ .

Reduction of the intensity data was carried out in a manner similar to that for 5,6-dihydrodibenz[*a,j*]anthracene (Wei, 1972). The absorption corrections (Busing & Levy, 1957) were calculated with a linear absorption coefficient of  $5.49 \text{ cm}^{-1}$  for Cu  $K\alpha$  radiation; the maximum and minimum transmission factors were 0.981 and 0.940 respectively. Of 1003 independent reflections measured, 854 had observed intensities greater than zero and were subsequently utilized as the 'observed' data. The reflections with *k* odd for *0kl*, *l* odd for *h0l*, and *h* odd for *hk0* were mostly weak, indicating pseudosymmetry of space group *Pbca* ( $D_{2h}^{15}$ , No. 61). These reflections constitute only 13.2% of the total number of observed data (113 out of the 854 observed).

### Solution and refinement of the structure

The close agreement of the cell parameters of the present compound with those for the orthorhombic modification of the parent compound, dibenz[*a,h*]anthra-

cene† (or 1:2:5:6-dibenzanthracene) (Robertson & White, 1947), suggests that the two compounds have similar structures. It follows that the molecule of the present compound is centered near the origin of the unit cell, with the symmetry reduced from *Pbca* to  $P2_12_12_1$ , because of the lack of a crystallographic center of symmetry.

The structure was solved by applying the symbolic-addition phase-determination procedure (Karle & Karle, 1966), using programs *FAME* and *MAGIC* written by Dewar & Stone (1967). A three-dimensional *E* map was first calculated (program *FORDAPER* of A. Zalkin as modified by G. Brunton), based on space group *Pbca* using 127 reflections. The positional and thermal parameters for the 11 carbon atoms in the asymmetric unit were refined (using only the 741 pseudocentric data) by the full-matrix least-squares method (Busing, Martin & Levy, 1962) in which the function minimized was  $\sum w(F_o^2 - s^2 F_c^2)^2$ , where *s* is the scale factor, and the weights *w* are equal to the reciprocals of the variances  $\sigma^2(F_o^2)$ . The unweighted reliability index,  $R(F^2) = [\sum |F_o^2 - s^2 F_c^2| / \sum F_o^2] \times 100$ , was reduced to 17.0% with isotropic and 14.5% with anisotropic treatment of the thermal motions. At this stage, the positions of 11 carbon

\* There was no sign of any diffuseness of the spectra, nor any other irregularity in the diffraction patterns, which appeared identical for several crystals obtained from two batches. Furthermore, the variation of the mosaicity in different directions was not more than  $1^\circ$ .

† Crystal data reported were  $a = 8.22$ ,  $b = 11.39$ ,  $c = 15.14 \text{ \AA}$ ,  $V = 1417.9 \text{ \AA}^3$ , space group *Pcab*. This space group differs from the standard choice of *Pbca* only by an interchange of the *b* and *c* axes (*International Tables for X-ray Crystallography*, 1965, p. 548). The molecule is centered at a crystallographic center of symmetry.

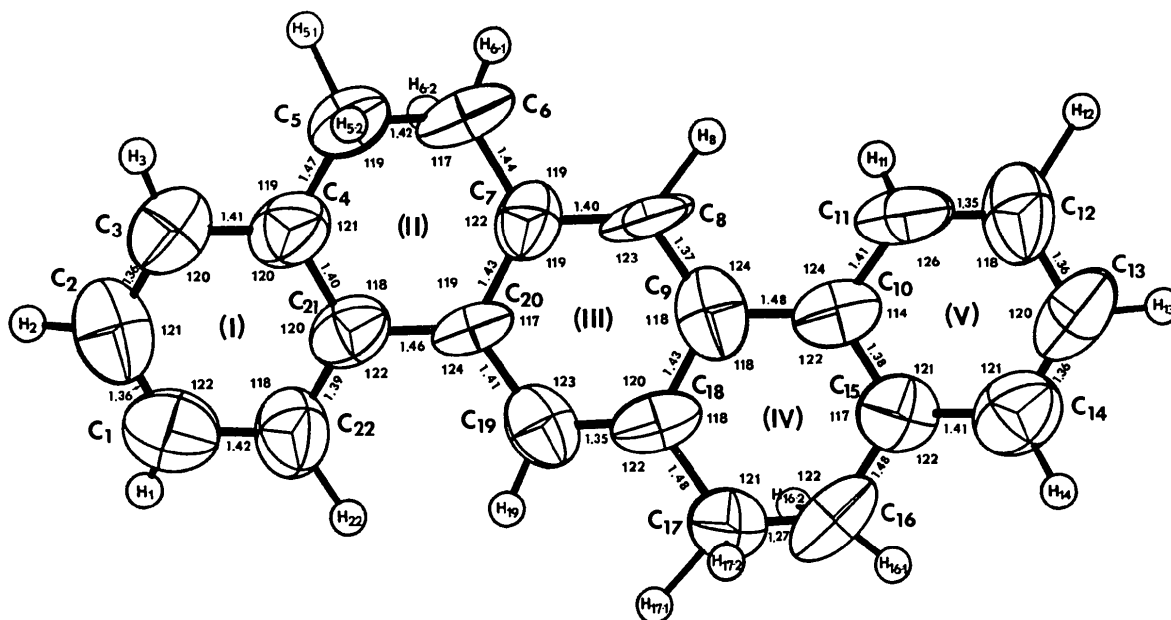


Fig. 1. Configuration of the 5,6-dihydrodibenz[*a,h*]anthracene molecular unit (the average molecule), consisting of two nearly superimposable alternative orientations with an approximate occupancy factor ratio of 1:1. All averaged carbon atoms are shown with the 50% probability thermal ellipsoids. Standard deviations for C-C bond distances are mostly 0.01 Å, except for the bonds between C(13) and C(17), where they are 0.02 Å. The standard deviation for each C-C-C angle is  $1^\circ$ . The C-H bond distances range from 0.74 to 1.30 Å (with e.s.d.'s of 0.05–0.14 Å) except for two unusually large values: 1.42 (7) and 1.77 (13) Å, for C(12)-H(12) and C(16)-H(16-2) respectively. No particularly unusual bond angles involving hydrogen were found.

atoms, related to the original set by a center of symmetry at the origin, were added into the subsequent refinement in the true space group  $P2_12_12_1$ , using *all* the 854 observed data. Refinement with isotropic thermal motions yielded an  $R(F^2)$  value of 17.7%; with anisotropic thermal motions, 13.7%.

A three-dimensional difference Fourier map computed at this stage showed clearly the 10 hydrogen atoms attached to rings (I), (III), and (V) (see Fig. 1). There were also two sets of four relatively lower hydrogen peaks of poor resolution, nearly related by the center of symmetry, one set being tetrahedrally attached to C(5) and C(6), and the other set to C(16) and C(17). Hydrogen atoms numbered with the suffix '-1' were equatorial, and those with the suffix '-2' were axial. Since the material used in this study was at least 99% pure (Lijinsky *et al.*, 1970), one would assume that two of the eight hydrogen positions were not real unless crystal disorder was present.

In an attempt to refine an ordered model, refinements were carried out for the molecule involving 22 carbon atoms and 16 hydrogen atoms, in one case with C(5) and C(6) assigned as tetrahedral and C(16) and C(17) assigned as aromatic, and *vice versa* in the other case. The refinements converged to give the closely similar  $R(F^2)$  values of 8.0 and 7.8% respectively. However, in each case the resulting bond distance between the carbon atoms assumed as saturated was  $\sim 0.1$  Å less than the normal single-bond distance. Furthermore, 'best' plane calculations (Smith, 1962) showed that while rings (I), (III), and (V) were nearly planar, rings (II) and (IV) were *both* nonplanar with out-of-plane deviations of the order of 0.1 Å for atoms C(5), C(6), C(16), and C(17), and that rings (I) and (V) were *both* twisted from ring (III) by angles of the order of  $10^\circ$ . We were therefore led to assume, on the basis of all the above evidence, that crystal disorder exists whereby molecules are randomly oriented head-to-tail or tail-to-head, so that the aromatic and partially saturated penultimate rings randomly occupy equivalent sites in the crystal.

Extensive efforts to refine several disordered models, by the use of a rigid-group refinement program (kindly supplied by Professor L. F. Dahl of the University of Wisconsin), were unsuccessful. Finally, the following model was adopted. Each of the 22 carbon atoms, the 10 hydrogen atoms on rings (I), (III), and (V), and the 4 equatorial hydrogen atoms on rings (II) and (IV) were represented by a single atom of occupancy factor 1. Each of these atomic positions would then represent the weighted average of two atomic sites in the two alternative configurations of the molecule. In addition, the two axial hydrogen atoms on ring (II) were given the same partial occupancy factor  $m$ , and the two axial hydrogen atoms on ring (IV) were each given the occupancy factor  $1-m$ .

A refinement was performed for this model, in which the thermal motions of the carbon atoms were treated anisotropically and the hydrogen atoms received in-

Table 1. Positional and thermal parameters

Entries  $X$ ,  $Y$ , and  $Z$  are the fractional coordinates multiplied by  $10^4$  for carbon atoms and  $10^3$  for hydrogen atoms. The form of the anisotropic temperature factor ( $\times 10^4$ ) is  $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ . For each hydrogen atom, entry  $\beta_{11}$  is the isotropic temperature factor in Å<sup>2</sup>. Standard deviations of the last significant figure(s) are given in parentheses here and in Table 3.

The occupancy factor  $m$  for H(5-2) was adjusted in addition to all other variable parameters in the least-squares refinement. The occupancy factor for H(6-2) was also  $m$  and the occupancy factors for H(16-2) and H(17-2) were each  $1-m$ . A value of  $m=0.58(5)$  was obtained. The temperature factors for H(5-2), H(16-2), and H(17-2) were set equal to that for H(5-2), which was adjusted.

| ATOM    | X         | Y         | Z         | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---------|-----------|-----------|-----------|--------------|--------------|--------------|--------------|--------------|--------------|
| C(1)    | 576(15)   | -3399(9)  | 586(12)   | 421(32)      | 102(9)       | 152(14)      | 7(15)        | 34(17)       | -26(10)      |
| C(2)    | -126(15)  | -3580(8)  | 1611(10)  | 318(28)      | 70(7)        | 168(13)      | -10(13)      | -96(16)      | -11(9)       |
| C(3)    | -829(14)  | -2924(10) | 2235(9)   | 276(24)      | 92(8)        | 121(12)      | -30(12)      | -10(14)      | 13(8)        |
| C(4)    | -786(12)  | -2044(8)  | 1833(8)   | 249(21)      | 97(8)        | 107(9)       | 20(11)       | 11(11)       | 23(7)        |
| C(5)    | -1524(12) | -1342(9)  | 2421(10)  | 228(21)      | 86(7)        | 150(13)      | -16(11)      | 69(14)       | 12(8)        |
| C(6)    | -1877(13) | -525(8)   | 1982(8)   | 231(21)      | 131(10)      | 78(9)        | -22(13)      | 53(11)       | 8(8)         |
| C(7)    | -964(11)  | -296(6)   | 974(7)    | 251(18)      | 63(5)        | 95(8)        | -7(9)        | -14(11)      | 15(5)        |
| C(8)    | -941(10)  | 586(7)    | 593(7)    | 214(18)      | 128(9)       | 73(7)        | 28(11)       | 82(9)        | 18(7)        |
| C(9)    | -49(12)   | 859(6)    | -324(8)   | 258(20)      | 63(5)        | 122(10)      | 2(10)        | -77(12)      | -2(6)        |
| C(10)   | -62(10)   | 1772(6)   | -788(7)   | 163(16)      | 95(7)        | 85(8)        | -17(10)      | 0(9)         | -11(6)       |
| C(11)   | -761(13)  | 2504(10)  | -228(8)   | 226(21)      | 127(9)       | 103(10)      | 24(13)       | 63(11)       | -2(8)        |
| C(12)   | -673(14)  | 3354(7)   | -575(9)   | 402(29)      | 76(6)        | 136(12)      | 18(12)       | -65(16)      | 28(8)        |
| C(13)   | 122(15)   | 3537(10)  | -1569(10) | 279(27)      | 114(10)      | 142(12)      | -26(14)      | 20(14)       | 54(9)        |
| C(14)   | 807(15)   | 2870(11)  | -2178(11) | 240(23)      | 107(10)      | 151(13)      | -6(13)       | 8(14)        | 15(9)        |
| C(15)   | 764(11)   | 1992(7)   | -1767(9)  | 157(17)      | 74(7)        | 146(11)      | -35(9)       | -4(11)       | -16(7)       |
| C(16)   | 1492(20)  | 1256(12)  | -2417(10) | 599(47)      | 128(12)      | 175(16)      | -2(18)       | 170(24)      | 62(10)       |
| C(17)   | 1744(14)  | 500(7)    | -1961(9)  | 357(28)      | 80(7)        | 125(11)      | 38(12)       | 49(14)       | 6(7)         |
| C(18)   | 835(11)   | 206(8)    | -944(7)   | 176(15)      | 103(7)       | 73(8)        | -19(10)      | 14(9)        | -2(6)        |
| C(19)   | 861(11)   | -641(5)   | -566(8)   | 245(20)      | 55(5)        | 117(10)      | -4(9)        | -50(11)      | -23(6)       |
| C(20)   | 11(9)     | -931(6)   | 405(6)    | 160(15)      | 87(6)        | 69(7)        | -8(9)        | 41(8)        | 1(6)         |
| C(21)   | -97(10)   | -1898(7)  | 761(7)    | 201(17)      | 88(6)        | 85(8)        | -10(10)      | -6(9)        | 11(6)        |
| C(22)   | 566(13)   | -2534(8)  | 113(9)    | 340(26)      | 73(6)        | 131(11)      | 10(11)       | 25(13)       | 14(7)        |
| H(1)    | 81(8)     | -377(4)   | 8(5)      | 5(2)         |              |              |              |              |              |
| H(2)    | -53(8)    | -423(5)   | 179(5)    | 7(2)         |              |              |              |              |              |
| H(3)    | -124(5)   | -296(3)   | 303(4)    | 3(1)         |              |              |              |              |              |
| H(5-1)  | -241(8)   | -147(4)   | 332(5)    | 10(2)        |              |              |              |              |              |
| H(5-2)  | -86(16)   | -124(9)   | 285(11)   | 4(2)         |              |              |              |              |              |
| H(6-1)  | -226(6)   | -13(4)    | 253(4)    | 5(1)         |              |              |              |              |              |
| H(6-2)  | -299(12)  | -99(6)    | 155(8)    | 4            |              |              |              |              |              |
| H(8)    | -168(13)  | 120(7)    | 96(9)     | 17(4)        |              |              |              |              |              |
| H(11)   | -148(5)   | 239(3)    | 17(5)     | 2(1)         |              |              |              |              |              |
| H(12)   | -160(9)   | 403(4)    | -6(6)     | 11(2)        |              |              |              |              |              |
| H(13)   | -7(10)    | 417(5)    | -189(6)   | 9(2)         |              |              |              |              |              |
| H(14)   | 183(15)   | 305(6)    | -272(9)   | 21(5)        |              |              |              |              |              |
| H(16-1) | 194(8)    | 149(4)    | -296(5)   | 6(2)         |              |              |              |              |              |
| H(16-2) | -19(16)   | 75(7)     | -303(8)   | 4            |              |              |              |              |              |
| H(17-1) | 228(8)    | -24(4)    | -238(5)   | 9(2)         |              |              |              |              |              |
| H(17-2) | 304(17)   | 56(8)     | -171(11)  | 4            |              |              |              |              |              |
| H(19)   | 162(6)    | -108(3)   | -104(4)   | 2(1)         |              |              |              |              |              |
| H(22)   | 124(6)    | -232(3)   | -77(4)    | 6(1)         |              |              |              |              |              |

dividual isotropic temperature factors, except for the four axial hydrogen atoms, which were constrained to have the same isotropic temperature factor. At the end of the refinement, the values of  $R(F^2)$  and  $R_w(F^2)$  [the latter defined as  $(\sum w|F_o^2 - s^2F_c^2|/\sum wF_o^2)^{1/2} \times 100$ ] stood at 7.7 and 8.6% respectively. The unweighted reliability index based on  $F$  was 7.5%. The standard deviation of an observation of unit weight, defined as  $[\sum w|F_o^2 - s^2F_c^2|/(n-p)]^{1/2}$  (where  $n$  is the number of observations and  $p$  the number of parameters fitted to the data set), was 1.093. The parameter shifts in the last cycle for carbon atoms were all less than 53% of the corresponding standard deviations, whereas the maximum shift in hydrogen parameters was 95% of the corresponding standard deviation. The scattering factors used for carbon were those of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and those for hydrogen were of Stewart, Davidson & Simpson (1965). The final positional and thermal parameters are given in Table 1. The observed and calculated structure amplitudes are listed in Table 2. The bond lengths and



saturated carbon atoms in the structure of 5,6-dihydrodibenz[*a,j*]anthracene (Wei, 1972). While the C(5)–C(6) bond distance of 1.42 (1) Å is close to the occupancy-weighted average, 1.44 Å, the C(16)–C(17) bond distance of 1.27 (2) Å is 0.06 Å shorter than the aromatic distance. This anomaly cannot be explained, except as a consequence of the difficulty in treating the disorder problem. It is worth pointing out that in *averaged disordered* structures it is not uncommon to obtain chemically unrealistic bond distances and angles, and that individual molecular parameters based on least-squares refinement should be regarded as less certain than normal. [For examples, see the structures of Fe<sub>3</sub>(CO)<sub>12</sub> (Wei & Dahl, 1969) and Co<sub>4</sub>(CO)<sub>12</sub> (Wei, 1969).]

It is interesting that, in spite of the troublesome disorder problem, the two exceptionally long C–C bond distances – 1.48 (1) and 1.46 (1) Å for C(9)–C(10) and C(20)–C(21) respectively – are found to be compatible with 1.478 (4) and 1.459 (5) Å for the analogous bond distances in the structure of 5,6-dihydrodibenz[*a,j*]anthracene.

Two unusually short intramolecular H···H contacts of 2.02 (11) and 1.93 (7) Å, for H(8)···H(11) and H(19)···H(22), are much less than the accepted van der Waals contact of 2.4 Å (Pauling, 1960). These short contacts can be considered to be mainly responsible for the significant deviations of the four extra-annular C–C–C angles at C(9), C(10), C(20), and C(21) [ranging from 121.7 (9) to 124.3 (9)°] from the pure aromatic C–C–C angle of 120°.

The packing of the structure closely resembles that of the orthorhombic modification of its parent hydrocarbon, dibenz[*a,h*]anthracene (Robertson & White, 1947). Fig. 2 illustrates the arrangement of the molecules in the unit cell, viewed down the [100] direction. The intermolecular H···H contacts of less than 2.4 Å are

given in Table 3. Since some of these short contacts involve partial-hydrogen atoms on both sides of the molecule, one may speculate that they may be partly

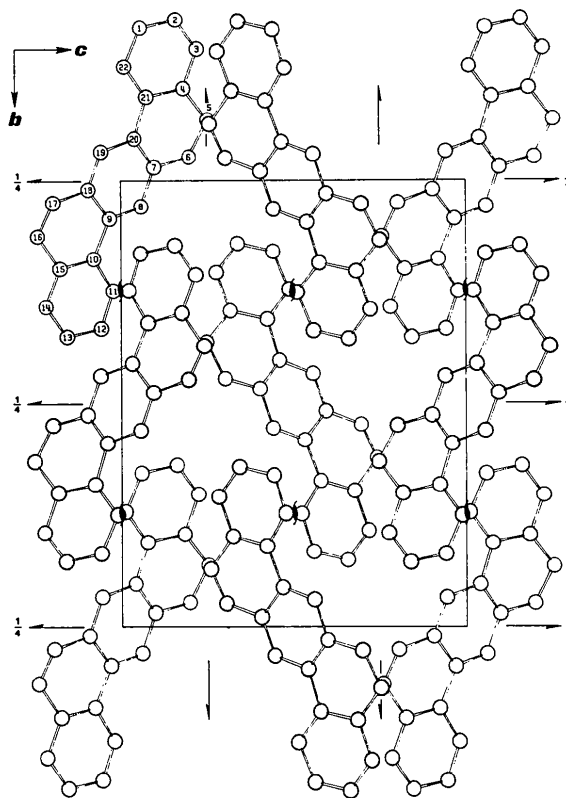


Fig. 2. Projection of the unit cell along the [100] direction. For the sake of clarity, hydrogen atoms are not included; only the basic molecule is labeled.

Table 3. Intermolecular H···H distances less than 2.4 Å\*

|                              |             |                                |             |
|------------------------------|-------------|--------------------------------|-------------|
| H(1)···H(6-2) <sup>i</sup>   | 2.18 (10) Å | H(12)···H(17-2) <sup>iii</sup> | 2.17 (14) Å |
| H(2)···H(17-1) <sup>ii</sup> | 2.13 (9)    | H(13)···H(17-1) <sup>iv</sup>  | 2.24 (10)   |
| H(2)···H(17-2) <sup>ii</sup> | 2.34 (14)   | H(13)···H(16-2) <sup>iv</sup>  | 2.39 (13)   |

\* Superscripts refer to the transformation of (*x,y,z*) into the following atomic positions:

|       |                    |                    |                    |
|-------|--------------------|--------------------|--------------------|
| i :   | $\frac{1}{2} + x$  | $-\frac{1}{2} - y$ | $-z$               |
| ii :  | $-\frac{1}{2} + x$ | $-\frac{1}{2} - y$ | $-z$               |
| iii : | $-\frac{1}{2} + x$ | $\frac{1}{2} - y$  | $-z$               |
| iv :  | $-x$               | $\frac{1}{2} + y$  | $-\frac{1}{2} - z$ |

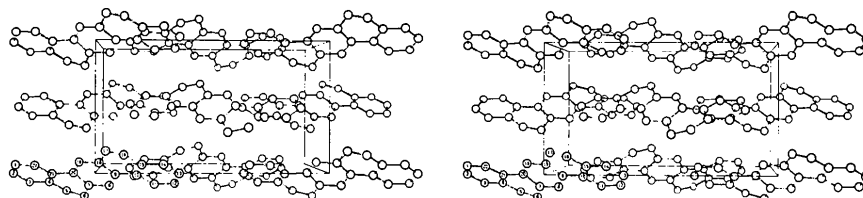


Fig. 3. Stereoscopic view of the unit cell down the *c* axis. The origin of the unit cell is in the lower left-front corner, and the *a* axis is vertical. Hydrogen atoms are not included; only the basic molecule is labeled.

responsible for the disorder. Fig. 3 shows a stereoscopic view down the  $c$  axis of the packing in the unit cell; for clarity, the hydrogen atoms are not included.

Because of the disorder in this structure, it seems impossible to obtain accurate parameters for an individual molecule, the most informative of which would be the torsion angle about the bond C(20)–C(21), where, in an individual molecule, ring (II) is partially saturated and ring (IV) is aromatic. This torsion angle relates two nearly planar moieties of the molecule: a 7-carbon moiety consisting of ring (I) plus atom C(5), and a 15-carbon moiety consisting of rings (III), (IV), and (V) plus atom C(6). In the closely related molecule of 5,6-dihydrodibenz[ $a,j$ ]anthracene (ordered molecule of the crystal structure), the equivalent torsion angle is  $21^\circ$ . A computer model-building program was used to show that torsion angles in the range  $15\text{--}21^\circ$  are possible. The present disordered structure appears to be consistent with this range, in that the torsion angle may be roughly estimated as the sum of the two apparent (averaged) torsion angles about bonds C(20)–C(21) and C(9)–C(10), or  $16.5^\circ$ . The analogous sum for the disordered molecule in the crystal of 5,6-dihydrodibenz[ $a,j$ ]anthracene is  $20^\circ$ .

We are indebted to Dr W. Lijinsky for furnishing the sample of the compound, and to Professor L. F. Dahl for making available the rigid-group refinement program. We are also grateful to Drs G. M. Brown and H. A. Levy of the Oak Ridge National Laboratory for their criticisms of the manuscript before it was submitted for publication.

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