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Conformation of Condensed Polycyclic Aromatic Hydrocarbons. I. The Molecular and Crystal Structure of Anthra[2,1,9,8-*hijkl*]benzo[*de*]naphtho[2,1,8,7-*stuv*]pentacene

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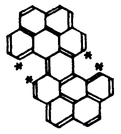
The crystal structure of the title compound, $C_{38}H_{18}$, has been determined by X-ray analysis. The space group is Pa, with unit cell dimensions a=31.276(4), b=3.7818(4), c=21.663(3) Å, β=124.22(1)° and Z=4. The structure was solved by the direct method and refined by full-matrix least-squares methods to an R value of 0.073 for 2619 reflections. Two crystallographically independent molecules are largely distorted from a planar structure due to the repulsions between the crowded hydrogen atoms, with different conformations to each other. The molecules are stacked face-to-face by van der Waals forces to form column structures along the b axis.

On treating naphthanthrone(6*H*-benzo[*cd*]pyren-6-one, hereafter abbreviated as NT) with copper powder, three condensed polycyclic aromatic hydrocarbons, diphenanthro[5,4,3-*abcd*:5′,4′,3′-*jklm*]perylene(DPP) (**1**), anthra[2,1,9,8-*ijklm*]benzo[*uv*]naphto[*defg*]pentaphene (**2**), and anthra[2,1,9,8-*hijkl*]benzo[*de*]naphtho[2,1,8,7-*stuv*]pentaphene (**3**) have been obtained.[1] All of these have eleven benzene rings. These hydrocarbons are stereochemically interesting, because they exhibit great steric hindrance between hydrogen atoms attached to carbon atoms indicated by asterisks, which is usually known as 1···7 interaction. They are also very important in the field of organic semiconductors due to their high photoconductivity.[2] However, the deficiency of information on their structures does not permit a detailed explanation of physical properties. The X-ray analysis was undertaken in order to elucidate the structure of anthra[2,1,9,8-*hijkl*]benzo[*de*]naphtho[2,1,8,7-*stuv*]pentaphene (**3**) and to compare its conformation with those of the related molecules.



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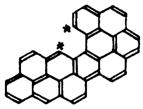

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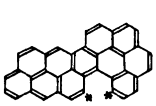

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Experimental

The compound was prepared by the method described in the previous paper,[1] and purified by recrystallization from chlorobenzene, chromatography on activated alumina, and sublimation in vacuum. The crystal used for X-ray work was grown from an *o*-dichlorobenzene solution. Preliminary unit cell dimensions and space group ($P2_1/a$) were obtained from oscillation and Weissenberg photographs. However, a careful reexamination of the Weissenberg photographs of b and c axes exposed for a long time (about 200 h) revealed that the 030 reflection had weak but significant intensity. Therefore, the true space group should be Pa. The unit cell dimensions were refined by the least-squares method on the basis of 2θ values measured on a Rigaku automated four-circle diffractometer (16 reflections, $2\theta \leqq 25°$). The crystal data are listed in Table 1. Intensity data up to $2\theta \leqq 125°$ were collected on the diffractometer with Ni-filtered Cu $K\alpha$ radiation (λ=1.5418 Å). An $\omega/2\theta$ scan technique was employed with a scan rate of 4° min^{-1} in 2θ. Three standard reflections were monitored after every 50 measurements; the net counts of these reflections did not change significantly over the period of data collection. A total of 2619 independent reflections, ($|F_o| \geqq 3\sigma(|F_o|)$), was used for the structure determination. Corrections for Lorentz and polarization factors were made, but not for absorption.

Structure Determination

The first trial structure was obtained by the MULTAN 78 program,[3] on the assumption of the space group $P2_1/a$. As the ten peaks occupying the centers of the benzene rings on the E-maps could be interpreted not to be false but true ones, we assumed that the crystal was disordered and molecules took alternative positions randomly. Refinement by the least-squares methods reduced the R value to 0.23. But further refinement was impossible on this model.

A new model with space group Pa was constructed from the old model with $P2_1/a$ by removing the crystallographic center of symmetry. The refinement was carried out by the block-diagonal least-squares method. After the R value converged to 0.133, the full-matrix least-squares program SHELX 76[4] was applied, where some rigid groups and bond lengths were under constraint. When the R value deduced to 0.102, the usual refinement without constraint were carried out by the block-diagonal least-squares method. The final R was 0.073 for 2619 observed reflections. No peaks higher than 0.4 $e\text{Å}^{-3}$ was found on the final difference map. The atomic scattering factors were taken from the International Tables for X-ray Crystallography.[5] The final atomic parameters and their estimated standard deviations are listed in Table 2. Lists of the ob-

Table 1. Crystal data

$C_{38}H_{18}$, *M.W.* 474.6, mp 362 °C	
$\lambda_{\max}^{\text{benzene}}$ = 533 nm	
Space group: Pa	
a=31.276(4) Å	U=2118.7(5) Å^3
b=3.7818(4) Å	Z=4
c=21.663(3) Å	D_x=1.488 g/cm^3
β=124.22(1)°	μ(Cu $K\alpha$)=26.4 cm^{-1}

TABLE 2. FINAL ATOMIC COORDINATES WITH THEIR ESTIMATED STANDARD DEVIATIONS, MULTIPLIED BY 10^4

Atom	*x*	*y*	*z*	*B* or B_{eq}/Å^2	Atom	*x*	*y*	*z*	*B* or B_{eq}/Å^2
C(1A)	−281(2)	1440(17)	3090(3)	2.8	C(1B)	5051(2)	−3561(20)	7161(4)	3.8
C(2A)	−482(2)	1338(18)	3530(4)	3.3	C(2B)	5234(3)	−3619(20)	6722(4)	4.2
C(3A)	−182(3)	2524(19)	4273(4)	4.0	C(3B)	4946(2)	−2349(20)	5991(4)	4.0
C(4A)	320(3)	3810(20)	4578(4)	4.0	C(4B)	4465(2)	−935(19)	5698(4)	3.5
C(5A)	524(3)	3914(20)	4143(4)	4.0	C(5B)	4252(2)	−821(16)	6127(3)	2.4
C(6A)	1019(2)	5357(17)	4439(3)	2.7	C(6B)	3740(2)	443(18)	5849(3)	3.4
C(7A)	1245(2)	5619(15)	4032(3)	1.7	C(7B)	3571(3)	526(22)	6307(4)	4.6
C(8A)	1743(2)	6947(17)	4313(3)	2.5	C(8B)	3073(3)	2141(19)	6022(4)	4.0
C(9A)	1928(2)	7061(16)	3856(3)	2.8	C(9B)	2864(2)	2214(18)	6422(3)	3.2
C(10A)	1618(3)	5835(19)	3112(4)	3.9	C(10B)	3134(2)	744(15)	7174(3)	1.8
C(11A)	1847(2)	5755(15)	2689(3)	1.8	C(11B)	2945(2)	745(20)	7631(4)	3.9
C(12A)	2407(3)	5903(21)	2970(4)	4.8	C(12B)	2395(2)	1343(13)	7312(3)	1.6
C(13A)	2790(2)	5273(16)	3729(3)	2.5	C(13B)	2004(2)	557(19)	6575(4)	3.6
C(14A)	3310(2)	6048(16)	4023(3)	2.5	C(14B)	1489(2)	1248(22)	6296(4)	4.6
C(15A)	3450(3)	7456(26)	3561(5)	6.0	C(15B)	1350(2)	2763(15)	6737(3)	2.1
C(16A)	3071(2)	8056(15)	2801(3)	1.8	C(16B)	1727(3)	3429(22)	7495(4)	5.2
C(17A)	3226(3)	9114(23)	2338(5)	5.4	C(17B)	1598(2)	4639(17)	8005(3)	2.8
C(18A)	2868(3)	9499(20)	1593(4)	4.1	C(18B)	1957(3)	4708(19)	8756(4)	3.9
C(19A)	2345(3)	8510(18)	1253(4)	3.3	C(19B)	2480(3)	3644(19)	9063(4)	3.8
C(20A)	1969(3)	8459(20)	484(4)	4.0	C(20B)	2835(3)	3440(20)	9849(4)	4.1
C(21A)	1486(3)	7026(20)	182(4)	4.2	C(21B)	3331(3)	2398(19)	10158(3)	3.5
C(22A)	1329(3)	5893(20)	652(4)	4.0	C(22B)	3492(2)	1237(17)	9683(3)	2.7
C(23A)	819(2)	4729(18)	331(3)	3.4	C(23B)	4003(2)	−16(20)	9986(3)	3.9
C(24A)	649(2)	3850(17)	788(3)	2.8	C(24B)	4150(3)	−1013(22)	9533(4)	4.6
C(25A)	987(2)	4127(20)	1570(4)	3.7	C(25B)	3810(2)	−715(15)	8739(3)	2.4
C(26A)	808(2)	3464(14)	2047(3)	1.9	C(26B)	3989(3)	−1388(20)	8263(4)	4.3
C(27A)	310(3)	2254(20)	1766(4)	4.4	C(27B)	4477(2)	−3028(15)	8530(3)	2.0
C(28A)	132(2)	1681(16)	2214(3)	2.8	C(28B)	4665(2)	−3108(18)	8079(3)	3.1
C(29A)	−375(3)	367(21)	1935(4)	4.3	C(29B)	5171(2)	−4688(17)	8380(3)	2.8
C(30A)	−581(2)	223(19)	2345(4)	3.9	C(30B)	5348(2)	−4781(18)	7937(4)	3.6
C(31A)	223(2)	2724(16)	3396(3)	2.7	C(31B)	4548(2)	−2107(17)	6867(3)	3.1
C(32A)	428(2)	2979(14)	2963(3)	1.7	C(32B)	4353(2)	−1990(19)	7332(4)	4.0
C(33A)	932(2)	4423(18)	3287(4)	3.7	C(33B)	3859(2)	−714(15)	7066(3)	2.0
C(34A)	1117(2)	4547(16)	2827(3)	2.4	C(34B)	3651(2)	−522(17)	7505(3)	2.8
C(35A)	1499(2)	5281(14)	1893(3)	1.8	C(35B)	3293(3)	435(19)	8417(4)	4.1
C(36A)	1670(2)	6158(17)	1435(3)	3.1	C(36B)	3135(2)	1377(17)	8905(3)	2.8
C(37A)	2181(2)	7468(18)	1721(4)	3.4	C(37B)	2621(2)	2474(16)	8585(3)	2.9
C(38A)	2549(2)	7273(13)	2505(3)	1.5	C(38B)	2255(3)	2594(22)	7786(4)	4.9

served and calculated structure factors, parameters of hydrogen atoms and thermal parameters for non-hydrogen atoms are kept in the office of the chemical society of Japan (Document No. 8250). The computation was done on the M-180 computer at Tokyo Institute of Technology and on the M-200H computer at the Computer Center of the University of Tokyo.

Results

Figure 1 illustrates the crystal structure viewed along the b axis. There exist two crystallographically independent molecules, A and B, in an asymmetric unit. Figure 2 shows the perspective drawing of the A molecule. A similar figure has been obtained for the B molecule. Molecules are largely distorted from a planar structure due to the repulsions between crowded hydrogen atoms. The numbering scheme is indicated in Fig. 3, together with the average values of the two corresponding bond lengths in the A and B molecules. Individual bond lengths and angles for each molecule are listed in Tables 3 and 4, respectively. The average C–C length of 1.413(10) Å is similar to 1.41(1) Å in NT,⁶⁾ 1.407(6) Å in 7*H*-benzo[*hi*]chrysen-7-one,⁷⁾ and 1.409(9) Å in tetrabenzo[*a,cd,j,lm*]perylene (TBP).⁸⁾ Although the values of the bond lengths and angles are not so reliable due to the pseudo symmetry described above, a similar distortion of the NT moiety is observed; the bond lengths of C(20)–C(21), C(36)–C(37), and C(11)–C(12) increases in that order (1.37→1.42→1.48 Å). They correspond to the sequences observed in TBP (1.348→1.418→1.474 Å) and DPP (1.37→1.43→1.49 Å).⁹⁾

Although the C–C–C bond angles range from 115°

to 129°, the three bonds around each carbon atom are nearly coplanar. The large difference in the bond angle C(10)–C(11)–C(12), 128.7° in A and 120.9° in B, will be discussed in the next section.

The molecules are stacked face-to-face by van der Waals forces to make column structures along the b axis. Figure 4 shows the overlapping mode of the A molecules; the situation for the B molecule is close to this. A list of the equations of several planes and their dihedral angles is deposited in the office of the Chemical Society of Japan. The deviations of the atoms from the mean plane of the whole molecule range from 0.92 to −0.60 Å. The interplanar spacings are 3.67 Å in A and 3.66 Å in B, which are

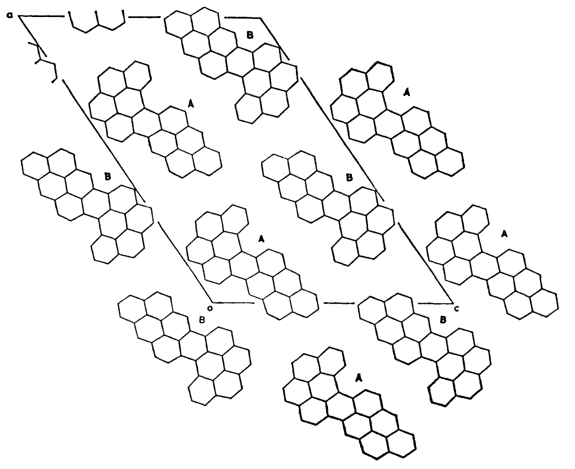


Fig. 1. A projection of the crystal structure along the b axis.

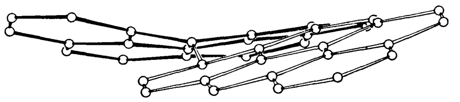


Fig. 2. A perspective drawing of the A molecule.

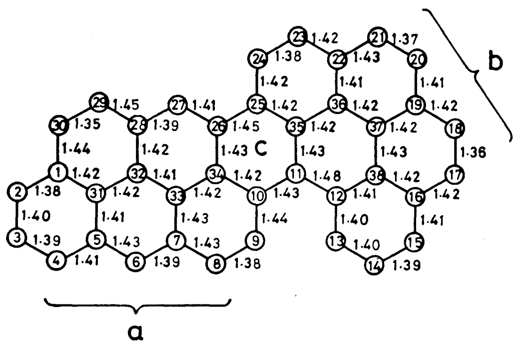


Fig. 3. The numbering scheme and mean values (*l*/Å) of bond lengths in the A and B molecules.

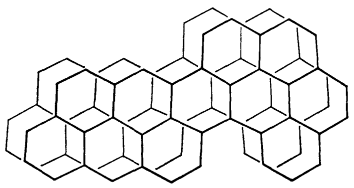


Fig. 4. Overlapping mode of the A molecules.

Table 3. Bond lengths

	A	B		A	B
	l/Å	*l*/Å		*l*/Å	*l*/Å
C(1)---C(2)	1.405	1.358	C(17)--C(18)	1.359	1.363
C(1)---C(30)	1.414	1.466	C(18)--C(19)	1.414	1.434
C(1)---C(31)	1.409	1.434	C(19)--C(20)	1.398	1.418
C(2)---C(3)	1.406	1.394	C(19)--C(37)	1.425	1.407
C(3)---C(4)	1.405	1.371	C(20)--C(21)	1.377	1.357
C(4)---C(5)	1.405	1.416	C(21)--C(22)	1.422	1.443
C(5)---C(6)	1.409	1.439	C(22)--C(23)	1.407	1.424
C(5)---C(31)	1.413	1.414	C(22)--C(36)	1.410	1.406
C(6)---C(7)	1.409	1.364	C(23)--C(24)	1.401	1.351
C(7)---C(8)	1.410	1.447	C(24)--C(25)	1.410	1.430
C(7)---C(33)	1.412	1.438	C(25)--C(26)	1.446	1.447
C(8)---C(9)	1.400	1.349	C(25)--C(35)	1.408	1.423
C(9)---C(10)	1.413	1.459	C(26)--C(27)	1.394	1.432
C(10)--C(11)	1.448	1.413	C(26)--C(34)	1.457	1.403
C(10)--C(34)	1.409	1.433	C(27)--C(28)	1.380	1.397
C(11)--C(12)	1.497	1.464	C(28)--C(29)	1.432	1.458
C(11)--C(35)	1.443	1.420	C(28)--C(32)	1.429	1.405
C(12)--C(13)	1.407	1.392	C(29)--C(30)	1.357	1.350
C(13)--C(38)	1.409	1.407	C(31)--C(32)	1.407	1.440
C(12)--C(14)	1.404	1.389	C(32)--C(33)	1.424	1.398
C(14)--C(15)	1.404	1.378	C(33)--C(34)	1.407	1.429
C(15)--C(16)	1.407	1.405	C(35)--C(36)	1.408	1.439
C(16)--C(17)	1.396	1.446	C(36)--C(37)	1.439	1.408
C(16)--C(38)	1.408	1.432	C(37)--C(38)	1.421	1.442

The e.s.d.'s range from 0.009 to 0.012 Å.

TABLE 4. BOND ANGLES

	A	B		A	B
	$\phi/°$	$\phi/°$		$\phi/°$	$\phi/°$
C(2)--C(1)--C(30)	120.3	123.6	C(23)-C(22)-C(36)	119.8	119.5
C(2)--C(1)--C(31)	119.9	119.0	C(22)-C(23)-C(24)	120.1	120.7
C(30)-C(1)--C(31)	119.7	117.4	C(23)-C(24)-C(25)	120.4	121.7
C(1)--C(2)--C(3)	120.2	122.2	C(24)-C(25)-C(26)	121.1	121.3
C(2)--C(3)--C(4)	119.9	120.0	C(24)-C(25)-C(35)	119.6	119.0
C(3)--C(4)--C(5)	120.2	120.4	C(26)-C(25)-C(35)	119.2	119.6
C(4)--C(5)--C(6)	120.1	123.9	C(25)-C(26)-C(27)	122.1	122.7
C(4)--C(5)--C(31)	119.9	119.1	C(25)-C(26)-C(34)	120.7	117.0
C(6)--C(5)--C(31)	120.0	117.0	C(27)-C(26)-C(34)	116.6	120.3
C(5)--C(6)--C(7)	123.4	119.9	C(26)-C(27)-C(28)	122.7	120.0
C(6)--C(7)--C(8)	125.2	117.6	C(27)-C(28)-C(29)	123.2	118.4
C(6)--C(7)--C(33)	115.1	125.1	C(27)-C(28)-C(32)	118.9	120.4
C(8)--C(7)--C(33)	119.7	117.3	C(29)-C(28)-C(32)	117.1	120.9
C(7)--C(8)--C(9)	120.1	121.9	C(28)-C(29)-C(30)	123.9	117.9
C(8)--C(9)--C(10)	120.3	121.5	C(1)--C(30)-C(29)	118.8	123.9
C(9)--C(10)-C(11)	117.6	125.0	C(1)--C(31)-C(5)	119.9	119.3
C(9)--C(10)-C(34)	119.6	118.0	C(1)--C(31)-C(32)	121.3	119.4
C(11)-C(10)-C(34)	122.7	116.7	C(5)--C(31)-C(32)	118.8	121.3
C(10)-C(11)-C(12)	128.7	120.9	C(28)-C(32)-C(31)	119.1	120.2
C(10)-C(11)-C(35)	116.7	120.0	C(28)-C(32)-C(33)	121.6	118.1
C(12)-C(11)-C(35)	114.6	118.9	C(31)-C(32)-C(33)	119.3	121.7
C(11)-C(12)-C(13)	120.0	123.4	C(7)--C(33)-C(32)	123.4	115.0
C(11)-C(12)-C(38)	119.4	118.2	C(7)--C(33)-C(34)	120.1	121.3
C(13)-C(12)-C(38)	119.7	118.3	C(32)-C(33)-C(34)	116.4	123.7
C(12)-C(13)-C(14)	120.2	121.1	C(10)-C(34)-C(26)	117.0	124.1
C(13)-C(14)-C(15)	120.1	121.0	C(10)-C(34)-C(33)	120.0	119.5
C(14)-C(15)-C(16)	120.0	120.1	C(26)-C(34)-C(33)	123.0	116.4
C(15)-C(16)-C(17)	118.9	122.6	C(11)-C(35)-C(25)	121.1	120.5
C(15)-C(16)-C(38)	119.8	118.5	C(11)-C(35)-C(36)	118.8	120.8
C(17)-C(16)-C(38)	121.1	118.7	C(25)-C(35)-C(36)	120.1	118.7
C(16)-C(17)-C(18)	119.7	121.5	C(22)-C(36)-C(35)	120.3	120.2
C(17)-C(18)-C(19)	122.0	120.5	C(22)-C(36)-C(37)	116.7	121.2
C(18)-C(19)-C(20)	125.2	119.4	C(35)-C(36)-C(37)	123.3	118.6
C(18)-C(19)-C(37)	118.4	119.6	C(19)-C(37)-C(36)	123.0	118.2
C(20)-C(19)-C(37)	116.5	120.7	C(19)-C(37)-C(38)	119.5	120.5
C(19)-C(20)-C(21)	122.4	120.9	C(36)-C(37)-C(38)	117.0	121.1
C(20)-C(21)-C(22)	120.4	119.8	C(12)-C(38)-C(16)	120.2	120.6
C(21)-C(22)-C(23)	119.5	121.6	C(12)-C(38)-C(37)	120.3	120.1
C(21)-C(22)-C(36)	120.5	118.9	C(16)-C(38)-C(37)	119.0	118.5

The e.s.d.'s range from 0.6 to 0.8°.

comparable to 3.84 Å in DPP.[9]

The molecules can also be regarded as two nearly planar NT skeletons connected by the central benzene ring. The deviations from the mean planes of the NT skeletons a, b are shown in Fig. 5. In both molecules, the planarity of (a) is better than that of (b). This brings about a wider interplanar spacing between (b)'s than that between (a)'s. Their values are 3.48 and 3.57 Å in A and 3.46 and 3.57 Å in B for (a) and (b) stackings, respectively. Each pair of NT skeletons in the A and B molecules makes an angle of 22.3° and an angle of 23.0°, respectively. The mean plane of the central benzene ring makes an angle of 5.7° with (a) and of 17.0° with (b) in A. The corresponding ones are 9.2° and 14.3° in B. Both central benzene rings take conformations of twisted

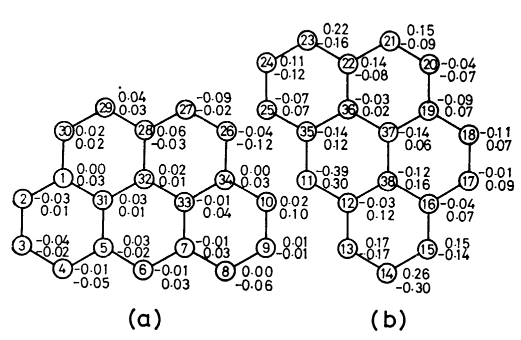


Fig. 5. Distances (l/Å) of atoms from the mean planes of (a) and (b) skeletons; upper and lower values of each pair refer to those in the A and B molecules, respectively.

boat form.

Discussion

The present study has established the structure of 3 and a large distortion from a planar structure occuring from the 1···7 interaction. Relief of such repulsion can be achieved by two modes of distortion as shown in Fig. 6; one is the enlargement of the C(3)–C(4)–C(5) angle, an angle-expansion mode, and the other is the twisting of the two C–C bonds opposite to each other, a twist mode. We define the values α and β to estimate the degree of the distortion as shown in Fig. 6. They are closely related to each other to avoid the 1···7 repulsion; β decreases with the increase of $(\alpha-120°)$.

In the present molecules, α and β of A are 128.7° and 30.1°, wheareas those of B are 120.9° and 37.6°, respectively. These values are significantly different for the two molecules. The repulsion has been relieved to equal extent, since the distance of C(9)···C(13) is 2.94 Å in A and 2.96 Å in B.

Four related molecules have been reported, as shown in Fig. 7, where the asterisks indicate the 1···7 interaction. The values of α and β, and the non-bonded distances between 2 and 6 carbon atoms are listed in Table 5. The four molecules are divided into two classes; an approximate two-fold axis exists perpendicular to the molecular plane in (1)—(3), whereas it lies in the molecular plane in (4). In each molecule of the former class, the two 1···7 repulsions may prevent the enlargement of α from 120° and should bring about the twisting mode. For the molecule of the latter class, on the other hand, only a repulsion may allow the angle-expansion mode to be taken.

Considering the values of α and β, the A and B molecules in the present crystal belong to the former and latter classes, respectively. The molecule has only a 1···7 repulsion between H(C9) and H(C13), but it has a fairly strong 1···6 repulsion between H(C24) and H(C27) opposite to the 1···7 repulsion. The distances between H(C24) and H(C27) are nearly the same as those of H(C9)···H(C13) for both molecules; 1.87(9) and 1.91(9) Å for A and 2.01(9) and 2.00(9) Å for B.[††] The present molecule may be described as an intermediate between the two classes. Consequently, both modes of distortion characteristic of the two classes can occur and the two molecular conformations will probably be observed in solution.

TABLE 5. COMPARISON OF STERIC HINDRANCES

Molecule	R		α/°	β/°	d(C···C)/Å
(1)a)	0.056	α_1)	123.5	41.0	2.971
		α_2)	122.7	41.5	2.944
(2)	0.28 (*hk*0)	α_1)	130	25	2.84
	0.28 (0*kl*)	α_2)	126	37	3.08
(3)b)	0.103	A)	119	39	3.01
		B)	121	30	2.87
This crystal	0.073	A)	128.7	30.1	2.94
		B)	120.9	37.6	2.96
(4)	0.23 (*hk*0)		131	32	3.01
	0.25 (0*kl*)				
	0.27 (*h*0*l*)				

a) The number of each molecule refers to that in Fig. 7. b) A and B indicate the crystallographically independent molecules which have symmetry C_2 perpendicular to molecular plane.

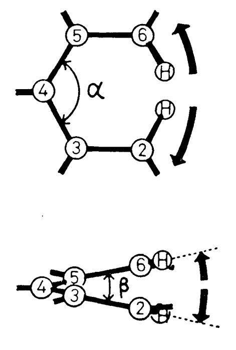
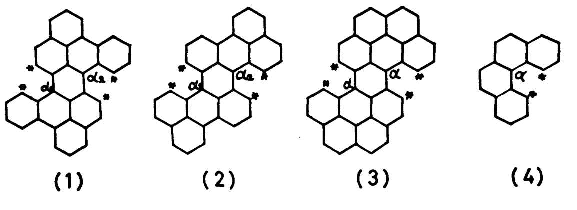


Fig. 6. Relief modes of 1···7 interaction.



(1) (2) (3) (4)

Fig. 7. (1): Tetrabenzo[*a,cd,j,lm*]perylene,[8] (2): tetrabenzo[*de,hi,op,st*]pentacene,[10] (3): diphenanthro[5,4,3-*abcd*:5′,4′,3′-*jklm*]perylene,[9] (4): benzo[*c*]phenanthrene.[11]

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[††] The distances of H···H based on the coordinates derived from an idealized geometry are 1.87 (H(C9)···H(C13)) and 1.87 Å (H(C24)···H(C27)) in A, and those of B are 1.96 and 2.02 Å. These agree with the observed values within the estimated standard deviations.