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

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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*; μ(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 $2\theta_{\max.} = 50°$ 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

Table 2. *Fractional atomic coordinates* (×10E) *and thermal parameters* (Å^2 ×10^{-3}) *and their standard deviations*

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2866 (5)	8000 (3)	−491 (7)	50 (2)	119 (3)	60 (2)	10 (2)	13 (2)	8 (2)
C(2)	3840 (5)	8529 (3)	557 (7)	50 (2)	88 (2)	64 (2)	13 (2)	17 (2)	9 (2)
C(3)	4794 (5)	7992 (2)	1589 (6)	40 (1)	68 (2)	50 (2)	4 (1)	21 (1)	1 (1)
C(4)	6117 (5)	8077 (2)	2819 (6)	49 (2)	55 (2)	46 (2)	2 (1)	13 (1)	−1 (1)
C(5)	7317 (5)	8350 (2)	2164 (6)	46 (2)	55 (2)	36 (1)	−2 (1)	6 (1)	4 (1)
C(6)	7817 (5)	9219 (3)	1857 (6)	62 (2)	53 (2)	51 (1)	0 (2)	6 (2)	6 (1)
C(7)	8935 (6)	9235 (3)	1153 (6)	63 (2)	72 (2)	60 (2)	−18 (2)	15 (2)	10 (2)
C(8)	9524 (5)	8414 (3)	768 (6)	47 (2)	82 (2)	57 (2)	−10 (2)	13 (2)	4 (2)
C(9)	9020 (6)	7500 (0)	1080 (6)	36 (2)	67 (3)	44 (3)	0 (0)	7 (2)	0 (0)
C(10)	7913 (6)	7500 (0)	1778 (6)	37 (2)	56 (2)	37 (2)	0 (0)	6 (2)	0 (0)
H(1)	2109 (32)	8394 (30)	−1347 (47)	44	144	90	18	3	9
H(2)	3828 (34)	9231 (26)	517 (42)	59	92	74	27	15	11
H(4)	6122 (30)	8424 (21)	3871 (36)	64	60	44	6	20	−2
H(6)	7425 (32)	9842 (23)	2028 (34)	74	52	61	0	12	7
H(7)	9269 (30)	9867 (28)	919 (35)	72	78	78	−22	16	17
H(8)	10284 (32)	8389 (24)	212 (32)	47	107	76	−13	22	9
C(11)	517 (6)	1986 (6)	3927 (7)	50 (2)	387 (15)	68 (3)	−24 (4)	6 (2)	17 (4)
C(12)	1478 (7)	1449 (6)	3386 (7)	76 (3)	267 (7)	64 (3)	−56 (4)	−6 (2)	26 (4)
C(13)	2425 (5)	1996 (3)	2828 (6)	54 (2)	176 (4)	41 (2)	−32 (2)	1 (2)	11 (2)
C(14)	3667 (6)	1922 (3)	2142 (6)	75 (2)	99 (2)	38 (2)	−6 (2)	13 (2)	−5 (2)
C(15)	4941 (5)	1661 (2)	3241 (6)	69 (2)	75 (2)	48 (2)	−1 (2)	28 (2)	−2 (2)
C(16)	5507 (6)	785 (3)	3711 (7)	95 (3)	87 (3)	76 (2)	1 (2)	41 (2)	0 (2)
C(17)	6746 (6)	771 (4)	4816 (7)	95 (3)	108 (3)	85 (6)	18 (2)	36 (2)	16 (2)
C(18)	7397 (6)	1592 (4)	5459 (7)	64 (2)	125 (3)	69 (2)	24 (2)	23 (2)	18 (2)
C(19)	6840 (6)	2500 (0)	4985 (7)	61 (3)	109 (4)	58 (3)	0 (0)	29 (2)	0 (0)
C(20)	5597 (6)	2500 (0)	3841 (7)	49 (2)	78 (3)	43 (2)	0 (0)	24 (2)	0 (0)
H(11)	−336 (42)	1674 (45)	4341 (57)	62	432	108	−65	24	15
H(12)	1293 (46)	705 (38)	3413 (47)	113	240	84	−96	13	11
H(14)	3492 (39)	1548 (27)	1147 (38)	112	119	40	−20	20	−5
H(16)	5072 (44)	99 (30)	3168 (45)	165	84	87	−12	34	6
H(17)	7217 (43)	35 (32)	5141 (48)	153	112	115	47	34	29
H(18)	8274 (36)	1482 (31)	6249 (49)	64	160	95	30	22	20
C(21)	−792 (5)	6998 (4)	6208 (6)	51 (3)	228 (6)	60 (2)	−15 (2)	16 (2)	6 (2)
C(22)	121 (6)	6468 (4)	5590 (7)	58 (2)	155 (4)	67 (2)	−31 (2)	1 (2)	5 (2)
C(23)	1021 (5)	7001 (3)	4979 (6)	49 (2)	116 (3)	46 (2)	−16 (2)	1 (1)	2 (2)
C(24)	2237 (5)	6925 (3)	4252 (6)	54 (2)	95 (2)	47 (2)	−11 (2)	14 (2)	−12 (2)
C(25)	3529 (5)	6654 (2)	5375 (6)	55 (2)	65 (2)	46 (2)	−4 (1)	20 (1)	−4 (1)
C(26)	4092 (6)	5787 (3)	5871 (7)	79 (2)	57 (2)	75 (2)	−0 (2)	36 (2)	−1 (2)
C(27)	5310 (6)	5763 (3)	7001 (7)	84 (2)	72 (2)	73 (2)	19 (2)	31 (2)	14 (2)
C(28)	5949 (5)	6583 (3)	7635 (6)	54 (2)	95 (3)	54 (2)	17 (2)	15 (2)	14 (2)
C(29)	5412 (6)	7500 (0)	7130 (7)	50 (2)	77 (3)	41 (2)	0 (0)	15 (2)	0 (0)
C(30)	4197 (5)	7500 (0)	5984 (6)	38 (2)	65 (3)	44 (2)	0 (0)	15 (2)	0 (0)
H(21)	−1501 (39)	6701 (39)	6670 (50)	49	290	80	−35	16	−0
H(22)	106 (39)	5757 (33)	5633 (49)	82	169	86	−63	19	−11
H(24)	2115 (33)	6609 (26)	3235 (40)	72	113	46	−16	12	−16
H(26)	3600 (29)	5223 (25)	5359 (45)	116	59	101	−10	37	−8
H(27)	5724 (37)	5127 (28)	7378 (43)	111	87	111	39	38	32
H(28)	6775 (34)	6563 (27)	8406 (44)	51	138	65	22	16	17
C(31)	7216 (5)	2997 (3)	10706 (6)	47 (1)	86 (1)	53 (2)	−2 (1)	11 (1)	−1 (1)
C(32)	6259 (5)	3528 (3)	9649 (6)	55 (2)	60 (2)	59 (2)	−11 (1)	21 (1)	−3 (1)
C(33)	5315 (5)	3000 (2)	8600 (6)	44 (1)	60 (1)	40 (1)	−4 (1)	17 (1)	−2 (1)
C(34)	4048 (5)	3072 (2)	7285 (6)	59 (2)	53 (1)	35 (1)	1 (1)	16 (1)	3 (1)
C(35)	2788 (5)	3343 (2)	7779 (5)	47 (1)	57 (2)	34 (1)	5 (1)	7 (1)	3 (1)
C(36)	2238 (6)	4218 (3)	8024 (6)	63 (2)	64 (2)	50 (2)	8 (1)	16 (1)	3 (1)
C(37)	1058 (5)	4242 (3)	8583 (6)	65 (2)	76 (2)	61 (2)	17 (2)	20 (2)	−2 (2)
C(38)	416 (5)	3422 (3)	8891 (6)	48 (2)	100 (3)	55 (2)	10 (2)	15 (1)	−8 (3)
C(39)	944 (6)	2500 (0)	8624 (6)	43 (2)	83 (3)	38 (2)	0 (0)	7 (2)	0 (0)
C(40)	2127 (5)	2500 (0)	8066 (6)	41 (2)	64 (3)	30 (2)	0 (0)	4 (2)	0 (0)
H(31)	7916 (32)	3333 (25)	11491 (41)	51	104	64	−13	8	−11
H(32)	6224 (33)	4199 (24)	9679 (39)	65	65	70	−16	19	−7
H(34)	4183 (29)	3377 (24)	6346 (38)	65	71	42	−0	19	11
H(36)	2667 (32)	4833 (25)	7782 (37)	80	58	79	13	18	9
H(37)	618 (34)	4898 (28)	8645 (39)	78	88	95	33	20	−4
H(38)	−459 (34)	3446 (25)	9170 (39)	52	120	73	17	20	−4

molecule and one disordered molecule. This model would not refine satisfactorily. A structure in *Pm* with equal numbers of identical molecules on the mirror planes gives systematic absences 0*k*0 with *k* odd, although there is no 2_1 axis. The *E* maps were successfully interpreted in terms of *Pm*; closer inspection of the diffractometer data showed one 0*k*0 reflexion (0,11,0) for which $I > 3\sigma(I)$.

Various constrained least-squares refinements were carried out, the results of which are summarized in Table 1. In the refinements where individual thermal parameters were refined, the U_{ij} values for the H atoms were kept fixed at those calculated from **T**, **L** and **S**. A scale factor and an isotropic extinction factor were included in the calculation. The minimum value of F_o/F_o (corr.) was 0·70. The final *R* was 0·032 for 458 parameters; R_w was 0·040. Atomic coordinates and thermal parameters are listed in Table 2.*

The thermal motion of the atoms was analysed, assuming that the molecule could be treated as a rigid body (Schomaker & Trueblood, 1968). **T**, **L** and **S** are given in Table 3. The value of **L** was used to correct the bond lengths for thermal motion.

Table 3. $\mathbf{T}_n$, $\mathbf{L}_n$ *and* $\mathbf{S}_n$ *and their estimated standard deviations relative to an orthogonal axial system with A parallel to a and C parallel to c**

T is in Å ×10^{-4}, **L** in (°) and **S** in (°) Å ×10^{-3}. The subscript *n* refers to the *n*th molecule. R.m.s. Δ, where $\Delta = U_{ij} - U_{ij}\text{(calc.)} = \delta_n$. $\delta_1 = 0{\cdot}0030, \delta_2 = 0{\cdot}0070, \delta_3 = 0{\cdot}0046, \delta_4 = 0{\cdot}0029$.

	M_{11}	M_{22}	M_{33}	M_{12}	M_{13}	M_{21}	M_{23}	M_{31}	M_{32}
$\mathbf{T}_1$	411 (9)	542 (12)	376 (14)	0	14 (9)		0		
$\mathbf{T}_2$	458 (20)	1052 (27)	347 (33)	0	111 (21)		0		
$\mathbf{T}_3$	439 (13)	711 (18)	385 (21)	0	43 (14)		0		
$\mathbf{T}_4$	440 (8)	542 (11)	300 (14)	0	56 (8)		0		
$\mathbf{L}_1$	8·8 (1·2)	5·7 (0·6)	11·2 (0·6)	0	0·2 (0·6)		0		
$\mathbf{L}_2$	18·3 (2·8)	9·2 (1·4)	33·9 (1·2)	0	5·3 (1·6)		0		
$\mathbf{L}_3$	20·9 (1·8)	3·6 (1·0)	20·4 (0·8)	0	−0·4 (1·1)		0		
$\mathbf{L}_4$	11·7 (1·2)	6·6 (0·6)	9·0 (0·6)	0	0·5 (0·6)		0		
$\mathbf{S}_1$	0	0	0	184 (25)	0	70 (17)	36 (11)	0	−56 (11)
$\mathbf{S}_2$	0	0	0	−453 (61)	0	−357 (39)	−70 (27)	0	−851 (28)
$\mathbf{S}_3$	0	0	0	−37 (40)	0	−143 (26)	41 (18)	0	−435 (19)
$\mathbf{S}_4$	0	0	0	−49 (25)	0	−85 (17)	−17 (11)	0	−125 (11)

Table 4. *Bond distances* (l_n), *and the corresponding values corrected for thermal vibration* (l_n^c)

The subscript *n* refers to the *n*th molecule, *e.g.* C(5) for the *n*th molecule is C[5 + (*n* − 1) × 10] in Table 2.

		l_1	l_2	l_3	l_4	l_1^c	l_2^c	l_3^c	l_4^c	$\langle l \rangle$	$\langle l^c \rangle$
m	C(1)–C(1)	1·386 (8)	1·424 (16)	1·390 (12)	1·377 (7)	1·390	1·436	1·399	1·381	1·386 (5)	1·392
j	C(1)–C(2)	1·394 (5)	1·398 (9)	1·391 (7)	1·391 (5)	1·398	1·408	1·397	1·394	1·393 (3)	1·398
i	C(2)–C(3)	1·382 (5)	1·402 (7)	1·379 (5)	1·378 (5)	1·386	1·412	1·385	1·382	1·383 (3)	1·389
l	C(3)–C(3)	1·363 (6)	1·397 (9)	1·383 (7)	1·384 (6)	1·367	1·408	1·392	1·388	1·379 (3)	1·386
h	C(3)–C(4)	1·524 (5)	1·527 (6)	1·524 (5)	1·524 (5)	1·528	1·537	1·529	1·528	1·525 (3)	1·530
k	C(4)–C(4)	1·598 (7)	1·602 (8)	1·594 (8)	1·586 (6)	1·603	1·615	1·604	1·591	1·594 (4)	1·602
g	C(4)–C(5)	1·516 (5)	1·472 (5)	1·498 (5)	1·496 (5)	1·520	1·479	1·504	1·500	1·496 (3)	1·501
a	C(5)–C(6)	1·358 (5)	1·365 (5)	1·359 (5)	1·374 (5)	1·362	1·375	1·367	1·378	1·364 (3)	1·371
b	C(6)–C(7)	1·417 (5)	1·403 (6)	1·398 (5)	1·401 (5)	1·421	1·410	1·403	1·404	1·405 (3)	1·410
c	C(7)–C(8)	1·364 (6)	1·370 (6)	1·362 (5)	1·369 (5)	1·368	1·380	1·369	1·373	1·367 (3)	1·373
d	C(8)–C(9)	1·417 (4)	1·403 (5)	1·412 (4)	1·426 (4)	1·421	1·413	1·422	1·430	1·416 (2)	1·423
e	C(9)–C(10)	1·404 (6)	1·423 (7)	1·403 (4)	1·404 (4)	1·408	1·429	1·429	1·407	1·406 (2)	1·411
f	C(15)–C(10)	1·402 (4)	1·383 (4)	1·398 (6)	1·400 (6)	1·406	1·392	1·406	1·404	1·395 (2)	1·401
n	C(1)–H(1)	1·09 (3)	1·11 (4)	1·00 (4)	0·99 (3)	1·09	1·11	1·00	0·99	1·55 (2)	1·05
o	C(2)–H(2)	0·97 (3)	1·05 (5)	0·99 (4)	0·93 (3)	0·98	1·06	0·99	0·93	0·98 (2)	0·98
p	C(4)–H(4)	1·04 (3)	0·99 (3)	0·97 (3)	0·96 (3)	1·04	1·00	0·98	0·97	0·99 (2)	1·05
q	C(6)–H(6)	0·98 (3)	1·11 (4)	0·98 (4)	1·00 (3)	0·98	1·12	0·98	1·00	1·01 (2)	1·02
r	C(7)–H(7)	0·98 (3)	1·14 (4)	1·00 (3)	1·02 (3)	0·98	1·14	1·00	1·02	1·03 (2)	1·03
s	C(8)–H(8)	1·01 (3)	1·01 (4)	0·95 (4)	0·98 (3)	1·01	1·01	0·95	0·98	0·99 (2)	0·99

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* A list of structure factors and a table of observed and calculated values of α and β have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32036 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Bond distances corrected for thermal motion are given in Table 4, angles in Table 5, and short intermolecular distances in Table 6. The numbering of atoms and the labelling of bonds are shown in Fig. 1.

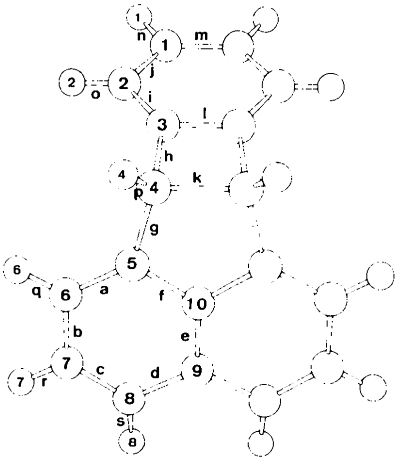

Fig. 1. Molecule 1 showing the numbering of the atoms and the labelling of the bonds. The corresponding atoms in the other molecules are numbered so that $n_2 = n_1 + 10$ for the second molecule, $n_3 = n_1 + 20$, *etc*. The large circles represent C atoms, the small ones H atoms.

Computational details

Calculations were carried out on a CDC6400 computer with the following programs: data reduction, *DATAP* and *DSORTH* (State University of New York at Buffalo); Fourier syntheses, *ZALKINS* (A. Zalkin, Lawrence Radiation Laboratory); constrained refinement, *KONSLS* (Pawley, 1971); conventional least-squares refinement, *LINUS* (Coppens & Hamilton, 1970); distances and angles, *ORFFE* (Busing, Martin & Levy, 1964); drawings, *ORTEP* (Johnson, 1965); energy minimization, *STRAIN* (Hazell, 1976*b*).

The quantity minimized was $\Sigma\, w(|F_o| - |F_c|)^2/\Sigma\, w|F_o|^2$, where $w = \{[\sigma(F_o^2) + 1{\cdot}03F_o^2]^{1/2} - |F_o|\}^{-2}$. The scattering factor of Cromer & Mann (1968) was used for C, and that of Stewart, Davidson & Simpson (1965) for H. The atom–atom potentials were those of Dashevski, Struchkov & Akopyan (1966), and the elastic constants those of Dashevski & Kitaigorodsky (1967).

Table 5. *Angles* (θ_n) *and the calculated values* ($\theta_{\text{calc.}}$)

The subscript *n* refers to the *n*th molecule. The dihedral angle is the angle at the hinge.

		θ_1	θ_2	θ_3	θ_4	$\langle\theta\rangle$	$\theta_{\text{calc.}}$
jm	C(1)–C(1)–C(2)	121·7 (2)	122·1 (4)	121·9 (3)	122·0 (2)	121·86 (12)	121·6
ij	C(1)–C(2)–C(3)	115·8 (4)	115·2 (7)	115·7 (5)	115·9 (3)	115·79 (21)	116·4
il	C(2)–C(3)–C(3)	122·5 (2)	122·7 (4)	122·4 (3)	122·1 (2)	122·38 (12)	122·0
hl	C(3)–C(3)–C(4)	94·4 (2)	93·8 (2)	94·0 (2)	93·8 (2)	94·01 (10)	94·0
hi	C(2)–C(3)–C(4)	142·7 (3)	143·4 (5)	143·6 (4)	144·0 (3)	143·40 (18)	144·0
hk	C(4)–C(4)–C(3)	85·6 (2)	86·2 (2)	86·0 (2)	86·2 (2)	85·99 (10)	86·0
gh	C(3)–C(4)–C(5)	114·3 (3)	116·6 (3)	114·8 (3)	115·6 (3)	115·33 (15)	117·8
gk	C(4)–C(4)–C(5)	104·5 (2)	104·2 (2)	104·5 (2)	104·5 (2)	104·43 (10)	104·3
ag	C(4)–C(5)–C(6)	132·1 (3)	131·5 (4)	132·3 (3)	132·5 (3)	132·18 (16)	132·0
fg	C(4)–C(5)–C(10)	108·3 (3)	108·4 (3)	108·5 (3)	108·9 (3)	108·54 (15)	109·0
af	C(10)–C(5)–C(6)	119·6 (3)	120·1 (3)	119·2 (3)	118·5 (3)	119·33 (15)	119·1
ab	C(5)–C(6)–C(7)	118·5 (3)	118·0 (4)	119·1 (4)	119·4 (4)	118·70 (18)	119·1
bc	C(6)–C(7)–C(8)	122·7 (4)	123·0 (4)	122·1 (4)	122·5 (4)	122·60 (20)	122·2
cd	C(7)–C(8)–C(9)	119·8 (3)	120·0 (4)	120·6 (4)	119·7 (3)	119·93 (17)	120·3
dd	C(8)–C(9)–C(8)	126·7 (4)	127·5 (6)	128·1 (5)	127·2 (5)	127·28 (24)	127·8
ff	C(5)–C(10)–C(5)	114·3 (4)	114·6 (4)	114·0 (4)	113·1 (4)	113·99 (20)	113·4
ef	C(5)–C(10)–C(9)	122·8 (2)	122·7 (2)	123·0 (2)	123·4 (2)	123·00 (10)	123·2
de	C(8)–C(9)–C(10)	116·7 (2)	116·2 (3)	115·9 (2)	116·4 (2)	116·32 (11)	116·1
Dihedral angle		114·0 (3)	116·4 (3)	114·6 (3)	115·6 (3)	115·16 (15)	117·4
mn	C(1)–C(1)–H(1)	120·0 (2·2)	113·0 (3·3)	114·5 (3·1)	118·2 (2·1)	117·4 (1·3)	119·3
jn	C(2)–C(1)–H(1)	118·3 (2·2)	124·8 (3·4)	123·6 (3·2)	119·9 (2·1)	120·6 (1·3)	119·1
jo	C(1)–C(2)–H(2)	120·0 (2·2)	111·8 (2·7)	119·7 (2·4)	122·4 (2·1)	119·1 (1·2)	121·8
io	C(3)–C(2)–H(2)	124·2 (2·2)	132·9 (2·9)	124·5 (2·4)	121·6 (2·2)	125·0 (1·2)	121·9
hp	C(3)–C(4)–H(4)	119·0 (1·7)	112·6 (2·3)	118·0 (2·0)	114·3 (1·7)	116·3 (1·0)	115·1
kp	C(4)–C(4)–H(4)	117·6 (1·6)	121·4 (2·0)	116·7 (2·1)	116·0 (1·9)	117·9 (1·0)	114·6
gp	C(5)–C(4)–H(4)	112·5 (1·7)	113·3 (2·3)	113·2 (2·1)	116·2 (1·8)	113·9 (1·0)	115·1
aq	C(5)–C(6)–H(6)	124·5 (1·9)	122·4 (2·2)	115·4 (2·2)	120·2 (1·9)	120·9 (1·1)	121·0
bq	C(7)–C(6)–H(6)	117·0 (1·8)	119·4 (2·2)	125·4 (2·2)	120·4 (1·9)	120·2 (1·1)	120·0
br	C(6)–C(7)–H(7)	117·3 (2·1)	116·4 (2·3)	119·5 (2·3)	117·6 (2·1)	117·7 (1·1)	118·5
cr	C(8)–C(7)–H(8)	120·0 (2·1)	120·5 (2·3)	118·4 (2·3)	119·5 (2·1)	119·6 (1·1)	119·4
cs	C(7)–C(8)–H(8)	125·6 (2·0)	115·1 (2·5)	121·8 (2·4)	121·8 (2·1)	121·6 (1·2)	120·3
ds	C(9)–C(8)–H(8)	114·8 (2·0)	125·0 (2·6)	117·6 (2·4)	118·2 (2·1)	118·2 (1·2)	119·4

Discussion

The unit-cell contents are shown in Fig. 2. There are four independent half moleculess in the asymmetric unit; molecules 2 and 4 are on the mirror plane at $y=\frac{1}{4}$, molecules 1 and 3 at $y=\frac{3}{4}$. Molecules 1 and 4 are approximately related by a 2_1 axis through $x=\frac{1}{2}$, $z=\frac{1}{2}$. The two layers are not identical; in the layer at $y=\frac{1}{4}$ all the molecules point along $\bar{\mathbf{c}}$, in the layer at $y=\frac{3}{4}$ half point along $\bar{\mathbf{c}}$, and half along $\mathbf{c}$. In both layers the molecules are arranged so that the naphthalene of one molecule is parallel to the benzene of the other.

The constrained refinements show that the four molecules are not identical, the main differences being in the dihedral angles. Refinement with individual atomic temperature factors is also better than treating the molecule as a rigid body.

The bond lengths of the naphthalene and benzene fragments are similar to those for naphthalene (Pawley & Yeats, 1969) and benzene (Cruickshank, 1970). Bond *k* which is shared between a four- and a five-

Table 6. *Close approaches between molecules*; H···H *distances less than* 2·85, C···H *less than* 3·1 *and* C···C *less than* 3·70 Å

Symmetry code

(i)	$-1+x$,	y,	z	(ix)	x,	$\frac{1}{2}-y$,	$-2+z$
(ii)	x,	$-1+y$,	z	(x)	x,	$1\frac{1}{2}-y$,	$-2+z$
(iii)	x,	y,	$-1+z$	(xi)	$1+x$,	$1\frac{1}{2}-y$,	$-2+z$
(iv)	x,	$1+y$,	z	(xii)	x,	$1\frac{1}{2}-y$,	$1+z$
(v)	$1+x$,	$1+y$,	z	(xiii)	$1+x$,	$\frac{1}{2}-y$,	z
(vi)	x,	$\frac{1}{2}-y$,	z	(xiv)	$1+x$,	$1\frac{1}{2}-y$,	z
(vii)	$1+x$,	$\frac{1}{2}-y$,	$-2+z$	(xv)	$1+x$,	$\frac{1}{2}-y$,	$1+z$
(viii)	$-1+x$,	$1\frac{1}{2}-y$,	z				

C(5)···C(22^{xiv})	3·644	H(24)···C(8^{viii})	3·003
C(5)···C(21^{xiv})	3·654	H(27)···C(32)	2·943
C(6)···C(22^{xiv})	3·684	H(28)···C(8^{xii})	3·076
C(8)···C(24^{xiv})	3·646	H(28)···C(7^{xii})	3·068
C(8)···C(23^{xiv})	3·699	H(31)···C(11^{xv})	3·028
C(9)···C(23^{v})	3·619	H(34)···C(15^{vi})	2·998
C(11)···C(35^{vi})	3·654	H(1)···H(36^{x})	2·671
C(11)···C(40)	3·689	H(1)···H(37^{x})	2·811
C(13)···C(38^{ix})	3·629	H(2)···H(16^{iv})	2·663
C(14)···C(40^{iii})	3·644	H(2)···H(36^{x})	2·741
C(15)···C(31^{ix})	3·598	H(4)···H(16^{iv})	2·570
C(16)···C(26^{vi})	3·417	H(4)···H(17^{iv})	2·625
C(16)···C(27^{vi})	3·637	H(6)···H(32^{x})	2·517
C(17)···C(27^{vi})	3·411	H(6)···H(31^{x})	2·641
C(20)···C(31^{iii})	3·587	H(6)···H(17^{iv})	2·799
C(26)···C(36^{x})	3·677	H(6)···H(16^{iv})	2·828
H(1)···C(22^{x})	2·973	H(7)···H(37^{xi})	2·692
H(6)···C(17^{iv})	2·982	H(7)···H(38^{xi})	2·844
H(6)···C(16^{iv})	3·008	H(8)···H(37^{xi})	2·801
H(14)···C(40^{iii})	3·038	H(11)···H(31^{vii})	2·713
H(14)···C(36^{ix})	2·939	H(16)···H(26^{vi})	2·743
H(14)···C(35^{ix})	2·877	H(17)···H(27^{vi})	2·757
H(16)···C(4^{ii})	3·037	H(18)···H(38^{xiii})	2·589
H(16)···C(26^{vi})	3·037	H(22)···H(37)	2·833
H(17)···C(27^{vi})	3·023	H(26)···H(34)	2·717
H(18)···C(38^{xiii})	2·795	H(26)···H(36)	2·571
H(21)···C(28^{i})	2·921	H(27)···H(32)	2·347

Table 7. *Deviations from planarity* (Å $\times 10^{-3}$)

Negative deviations are away from the other half of the molecule. Mean displacements, angles between planes and torsion angles at the hinge are also given.

	Deviation, Δ				
Molecule	1	2	3	4	Mean
Plane 1					
C(1)	29	1	13	12	14
C(2)	−11	4	−5	−4	−4
C(3)	−53	−10	−24	−24	−28
C(4)	36	5	16	−16	10
Plane 2					
C(4)	27	20	28	29	26
C(5)	−24	−6	−14	−21	−16
C(6)	−21	−31	−28	−20	−25
C(7)	−2	−29	−13	5	−10
C(8)	17	22	27	21	22
C(9)	12	39	14	−2	16
C(10)	−7	7	−14	−26	−10
$\langle\lvert\Delta\rvert\rangle$	21·7	15·8	17·8	16·4	
Angle between planes	108·68°	114·90°	111·41°	112·05°	
Angle at hinge	113·99	116·45	114·59	115·49	

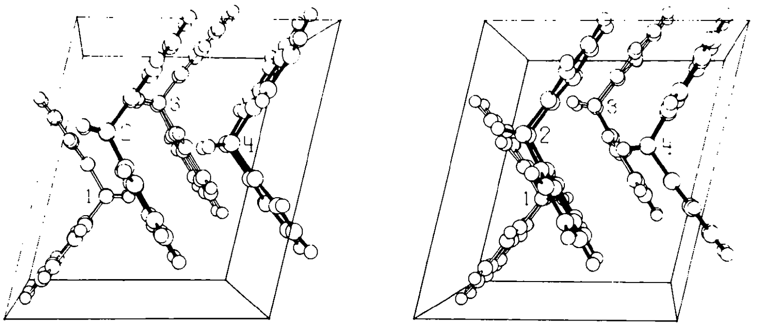

Fig. 2. The packing of the molecules as viewed along **b**; **a** is down the page, **c** across the page. The molecules at $y=\frac{1}{4}$ are shown with full bonds.

membered ring is very long, 1·606 (4) Å; the corresponding bonds in (I) [1·591 (6)] and (III) [1·601 (10) Å] are also long.

The joining of the five-membered ring to the naphthalene, and the four-membered ring to the benzene leads to strain which is propagated throughout the molecule. Energy-minimization calculations correctly reproduce the in-plane distortions but are not so good for the dihedral angle. The energy is insensitive to the dihedral angle, but for all three compounds the calculated dihedral angle is too large. The calculations were made assuming the two halves of the molecules to be planar. There are, however, small deviations from planarity (Table 7). The deviations are such as to make the dihedral angle at the hinge larger than the angle (φ) between the two best planes. The mean deviation from the plane, $\langle |\Delta| \rangle$, is proportional to $\varphi_{\text{calc.}} - \varphi$. There is a similar correlation between $\langle |\Delta| \rangle$ and the angle *gh*.

Although the bond distances of the benzene and the naphthalene fragments agree with those of the free molecules, the angles differ considerably, *e.g.* *ij* = 115·79 (120 in benzene), *dd* = 127·28 (121·6° in naphthalene). Energy calculations for a model with a 1,2-disubstituted benzene, the substituents being C atoms 1·5 Å from the benzene ring, predict that β should increase with α (see Fig. 3 for notation). Comparison with experimental values (Fig. 3) shows reasonable agreement between observed and calculated values.* A similar correlation might be expected between angles *gf* and *dd*; there is, however, a lack of experimental values for *gf*, other than ≃ 120 or ≃ 108°. The structures of 1,8-naphthobicyclobutane and of cyclohepta[*de*]naphthalene are being studied to provide data for *gf* > 120°.

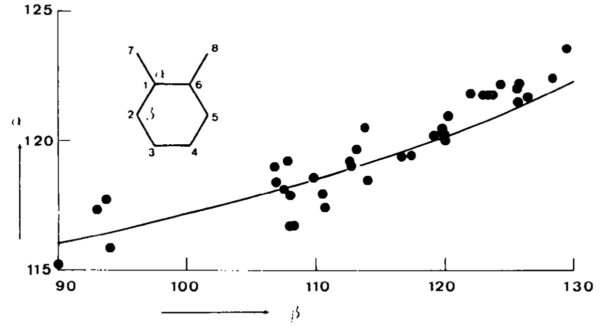

Fig. 3. Variation of α with β. The full circles (●) are observed values, the line represents calculated values. Calculations were made assuming C(1)–C(7) 1·50, C(1)–C(2) 1·395, C–H 1·08 Å.

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* See previous footnote.

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