

## Crystal and Molecular Structure of Bis-(2,2'-biphenylene)methane

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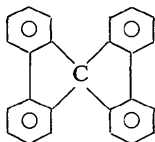
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Crystals of bis-(2,2'-biphenylene)methane are monoclinic of space group  $P2_1/c$  with cell constants  $a=10.173$ ,  $b=10.241$ ,  $c=16.722$  Å,  $\beta=96.54^\circ$ ,  $Z=4$ . The molecular symmetry is 1 as a result of the deviations from planarity of both biphenyl groups. The four central C-C-bonds are 1.53 Å, which is appreciably larger than the corresponding bond lengths in fluorene. The  $sp^2-sp^2$  single bonds in the biphenylene groups are 0.03 Å smaller than the corresponding bond in biphenyl itself.

### Introduction

The results of some investigations of intra-molecular triplet excitation transfer in the photoexcited triplet state of bis-(2,2'-biphenylene)methane (BBM),



randomly oriented in rigid glassy solution, has yielded detailed information about the rate and mechanism of this dynamic process (Shain, Ackerman & Teague, 1969). These results have also warranted an attempt to observe for the first time this phenomenon in the crystalline state (Shain & Weissman, 1971). For the interpretation the crystal structure of bis-(2,2'-biphenylene)methane is required and reported herein. The relation between structural details and the transfer of triplet excitation will be discussed by Shain & Weissman (1971).

### Experimental

Bis-(2,2'-biphenylene)methane crystallizes in the monoclinic system. The axes were chosen such that the space group is  $P2_1/c$  (extinctions  $h0l$  with  $l=2n+1$  and  $0k0$  with  $k=2n+1$ ). Cell constants were obtained by means of a least-squares procedure from  $h0l$  and  $0kl$ . Weissenberg pictures calibrated with Al-powder lines and taken with Cu  $K\alpha$  radiation at room temperature. They are  $a=10.173$  (2),  $b=10.241$  (2),  $c=16.722$  (2) Å,  $\beta=96.52$  (2)°. The density measured by flotation is  $1.19$  g.cm<sup>-3</sup>; the X-ray density with  $Z=4$  is  $1.21$  g.cm<sup>-3</sup>.

Intensities were measured with Cu  $K\alpha$  radiation with the help of a Nonius automatic single-crystal diffractometer using the moving-crystal moving-counter scanning method up to a maximum  $\theta$  of  $68.5^\circ$ . The dimensions of the crystal were  $0.3 \times 0.4 \times 0.5$  mm. Strong intensities were reduced by means of attenuation filters. The absorption correction was ignored.

Table 1. Fractional coordinates ( $\times 10^4$ ) and anisotropic thermal parameters (in Å<sup>2</sup>  $\times 10^3$ ) of the carbon atoms with their *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>u</i> <sub>11</sub>	<i>u</i> <sub>22</sub>	<i>u</i> <sub>33</sub>	2 <i>u</i> <sub>12</sub>	2 <i>u</i> <sub>23</sub>	2 <i>u</i> <sub>31</sub>
C(1)	3278 (4)	2139 (4)	1431 (3)	46 (2)	48 (2)	40 (2)	6 (4)	4 (4)	7 (3)
C(11)	3112 (5)	3504 (5)	1053 (3)	53 (3)	49 (2)	37 (2)	17 (4)	5 (4)	13 (4)
C(12)	4306 (5)	4158 (4)	1122 (3)	57 (3)	46 (2)	36 (2)	9 (4)	3 (4)	15 (4)
C(13)	4400 (6)	5419 (5)	814 (3)	75 (3)	50 (3)	54 (3)	12 (5)	16 (4)	31 (5)
C(14)	3258 (6)	5978 (6)	432 (4)	84 (4)	59 (3)	63 (3)	32 (6)	36 (5)	31 (6)
C(15)	2076 (7)	5326 (6)	358 (3)	81 (4)	73 (4)	58 (3)	59 (6)	29 (6)	9 (5)
C(16)	1968 (6)	4063 (6)	670 (3)	63 (3)	66 (3)	53 (3)	31 (5)	10 (5)	0 (5)
C(21)	4742 (4)	2143 (4)	1736 (2)	46 (2)	48 (2)	34 (2)	7 (4)	0 (3)	9 (3)
C(22)	5337 (4)	3318 (4)	1547 (3)	47 (2)	47 (2)	38 (2)	2 (4)	-1 (4)	12 (4)
C(23)	6667 (5)	3537 (5)	1775 (3)	52 (3)	55 (3)	56 (3)	-8 (4)	-5 (4)	6 (4)
C(24)	7400 (5)	2565 (6)	2201 (3)	49 (3)	69 (3)	57 (3)	-1 (5)	-5 (5)	-11 (4)
C(25)	6810 (5)	1405 (5)	2392 (3)	56 (3)	64 (3)	47 (2)	15 (5)	6 (5)	-5 (4)
C(26)	5482 (5)	1177 (5)	2156 (3)	54 (3)	52 (3)	45 (2)	5 (4)	16 (4)	8 (4)
C(31)	2374 (4)	1884 (5)	2084 (3)	41 (2)	50 (2)	48 (2)	14 (4)	11 (4)	12 (4)
C(32)	1507 (4)	869 (5)	1861 (3)	38 (2)	52 (3)	58 (3)	11 (4)	19 (4)	5 (4)
C(33)	580 (5)	475 (6)	2371 (4)	47 (3)	71 (3)	79 (4)	9 (5)	34 (6)	23 (5)
C(34)	583 (6)	1110 (7)	3108 (4)	57 (3)	88 (4)	74 (4)	35 (6)	44 (6)	48 (5)
C(35)	1485 (6)	2091 (6)	3332 (3)	67 (3)	79 (4)	56 (3)	39 (6)	12 (5)	38 (5)
C(36)	2380 (5)	2503 (5)	2824 (3)	61 (3)	62 (3)	49 (3)	14 (5)	0 (4)	26 (4)
C(41)	2835 (5)	1058 (5)	831 (3)	50 (3)	48 (2)	45 (2)	19 (4)	-4 (4)	-5 (4)
C(42)	1798 (5)	345 (5)	1082 (3)	43 (2)	49 (2)	57 (3)	9 (4)	3 (4)	-9 (4)
C(43)	1232 (6)	-677 (6)	614 (4)	59 (3)	56 (3)	76 (4)	7 (5)	-9 (5)	-23 (5)
C(44)	1724 (6)	-949 (6)	-105 (4)	73 (4)	59 (3)	77 (4)	23 (6)	-31 (6)	-32 (6)
C(45)	2754 (7)	-260 (6)	-352 (3)	89 (4)	75 (4)	53 (3)	50 (7)	-30 (6)	-9 (6)
C(46)	3348 (6)	774 (6)	115 (3)	70 (3)	70 (3)	48 (3)	31 (5)	-11 (5)	10 (5)

## Structure determination and refinement

The structure was determined by the symbolic addition method, using automatic computer programs (Schenk, 1969). The *E* Fourier synthesis of the solution with highest internal consistency revealed all 25 carbon atoms. The refinement was carried out by means of a block-diagonal least-squares program, applying the form factors from *International Tables for X-ray Crystallography*, (1962) and the weighting scheme of Cruickshank (1961). After some refinement cycles the hydrogen atoms were localized from a difference synthesis. Finally an *R* index of 6.0% for 2500 independent reflexions was obtained, refining positional and anisotropic thermal parameters of the carbon atoms and positional and isotropic thermal parameters of the hydrogen atoms. Approximately 20 reflexions were omitted in view of their systematic errors, which could either be caused by extinction or by dead-time measuring faults. A list of structure factors can be obtained from our laboratory, the lists of final parameters are given in Tables 1 and 2.

Table 2. Fractional coordinates ( $\times 10^3$ ) and isotropic thermal parameters (in  $\text{\AA}^2$ ) of the hydrogen atoms with their e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(13)	530 (6)	593 (7)	82 (4)	4 (1)
H(14)	332 (7)	683 (8)	24 (4)	5 (2)
H(15)	113 (8)	572 (8)	9 (5)	6 (2)
H(16)	97 (6)	353 (6)	64 (4)	4 (2)
H(23)	709 (6)	437 (6)	162 (4)	3 (1)
H(24)	837 (6)	274 (6)	235 (4)	3 (1)
H(25)	733 (7)	75 (8)	271 (5)	5 (2)
H(26)	507 (6)	35 (6)	230 (4)	3 (1)
H(33)	-21 (7)	-26 (7)	219 (4)	5 (2)
H(34)	-19 (6)	83 (6)	350 (4)	4 (1)
H(35)	150 (7)	255 (7)	386 (4)	5 (2)
H(36)	309 (6)	324 (6)	300 (4)	4 (1)
H(43)	31 (8)	-123 (8)	81 (5)	6 (2)
H(44)	146 (7)	-162 (6)	-47 (4)	4 (2)
H(45)	332 (7)	-39 (7)	-87 (4)	6 (2)
H(46)	415 (6)	137 (6)	-5 (4)	3 (1)

## Discussion

## Rigid body motion

The thermal parameters were used in a rigid body motion analysis (Schomaker & Trueblood, 1968), in which all 25 carbon atoms were assumed to form the rigid body. This supposition turned out to be justified judging by the factor  $[(\Delta U_{ij})^2/(m-s)]^{1/2} = 0.004 \text{ \AA}^2$ , which is approximately equal to the e.s.d.'s of the thermal parameters (see Table 1). The libration tensor *L* has principal axes 9.3, 5.1 and 5.1 ( $^\circ$ )<sup>2</sup>. The axis of largest libration (direction cosines 0.512, -0.701, 0.497) is nearly parallel to the plane through rings 3 and 4 and perpendicular to the plane through rings 1 and 2 (see Table 5). The libration tensor was used to calculate corrected bond lengths, which are given in Table 3.

## Accuracy

The accuracy of the determination can be estimated by assuming equivalent bond lengths to be independent

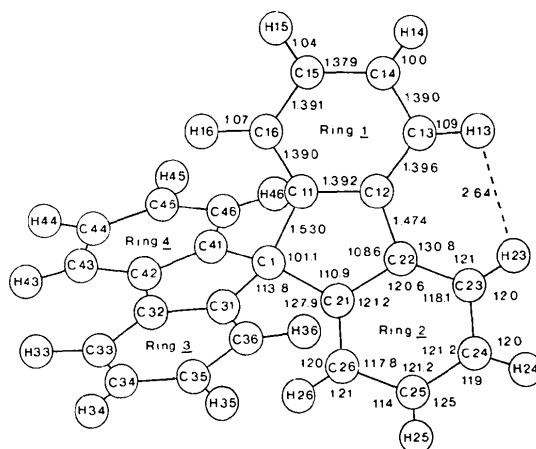


Fig. 1. Numbering of the atoms in bis-(2,2'-biphenylene)-methane. The mean values of the bond lengths and valence angles are indicated.

Table 3. Bond lengths ( $\text{\AA}$ ) of *BBM*

Corresponding bond lengths are given in rows together with their mean values, of which the experimental standard deviations are given in parentheses. The heading unc. refers to bond lengths calculated from the least-squares results, the heading corr. to bond lengths corrected for rigid body thermal motion. The e.s.d.'s are 0.008  $\text{\AA}$  for individual C-C bonds and 0.07  $\text{\AA}$  for C-H bonds.

Bond	Mean value		Ring 1		Ring 2		Ring 3		Ring 4	
	unc.	corr.	<i>i</i> =1 unc.	<i>j</i> =2 corr.	<i>i</i> =2 unc.	corr.	<i>i</i> =3 unc.	<i>j</i> =4 corr.	unc.	<i>i</i> =4 corr.
C( <i>i</i> 1)-C(1)	1.527 (3)	1.530 (3)	1.535	1.538	1.518	1.522	1.528	1.532	1.528	1.530
C( <i>i</i> 2)-C( <i>i</i> 1)	1.388 (3)	1.392 (3)	1.381	1.384	1.399	1.403	1.386	1.390	1.387	1.391
C( <i>i</i> 3)-C( <i>i</i> 2)	1.393 (4)	1.396 (4)	1.397	1.400	1.382	1.385	1.401	1.404	1.392	1.394
C( <i>i</i> 4)-C( <i>i</i> 3)	1.387 (2)	1.390 (2)	1.385	1.387	1.390	1.393	1.392	1.395	1.382	1.385
C( <i>i</i> 5)-C( <i>i</i> 4)	1.376 (5)	1.379 (5)	1.369	1.372	1.385	1.388	1.384	1.386	1.365	1.370
C( <i>i</i> 6)-C( <i>i</i> 1)	1.388 (1)	1.390 (1)	1.387	1.389	1.385	1.387	1.390	1.392	1.389	1.393
C( <i>i</i> 6)-C( <i>i</i> 5)	1.395 (8)	1.391 (8)	1.404	1.407	1.384	1.387	1.380	1.384	1.410	1.413
C( <i>j</i> 2)-C( <i>i</i> 2)	1.473 (2)	1.474 (2)	1.475	1.477			1.470	1.472		
H( <i>i</i> 3)-C( <i>i</i> 3)	1.08 (4)	1.09 (4)	1.05	1.06	0.99	1.00	1.12	1.12	1.17	1.18
H( <i>i</i> 4)-C( <i>i</i> 4)	0.99 (4)	1.00 (4)	0.93	0.94	1.00	1.01	1.11	1.12	0.93	0.94
H( <i>i</i> 5)-C( <i>i</i> 5)	1.04 (4)	1.04 (4)	1.09	1.09	.97	0.98	0.99	1.00	1.10	1.10
H( <i>i</i> 6)-C( <i>i</i> 6)	1.06 (4)	1.07 (4)	1.14	1.15	.98	0.99	1.06	1.07	1.07	1.08

measurements of the same quantity. This assumption proved to be correct, judging by the experimental standard deviations of equivalent bond lengths, which are approximately equal to the corresponding calculated e.s.d.'s. The e.s.d.'s are therefore a good estimate for the accuracy of the determination. This is also demonstrated by the bond angles (see Table 4). Fig. 1 shows the numbering of the atoms in BBM.

#### Bond lengths and angles

The bond lengths in the benzene ring are all normal within the limits of accuracy.

The mean of the bonds involving C(1) is 1.530 Å whereas Burns & Iball (1955) found a length of 1.47 (σ=0.02) for the corresponding bonds in fluorene. In fluorene, however, C(1) carries two hydrogen atoms, so that the difference in bond length, if real, may well be caused by hyperconjugation.

In biphenyl, Hargreaves & Rizvi (1962) and Trotter (1961) found a central bond of 1.506 Å. Hargreaves & Rizvi ascribed this lengthening of a single bond connecting *sp*<sup>2</sup> hybridized atoms to the repulsion of the *ortho* hydrogen atoms. In BBM no steric interaction between *ortho* hydrogen atoms is present as a result of the angle deformations at C(12) and C(22), so that the C(12)–C(22) bond has a normal length of 1.474 Å.

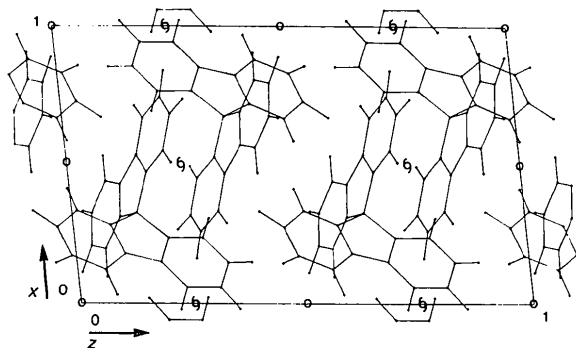


Fig. 2. Projection of the crystal structure along the *b* axis.

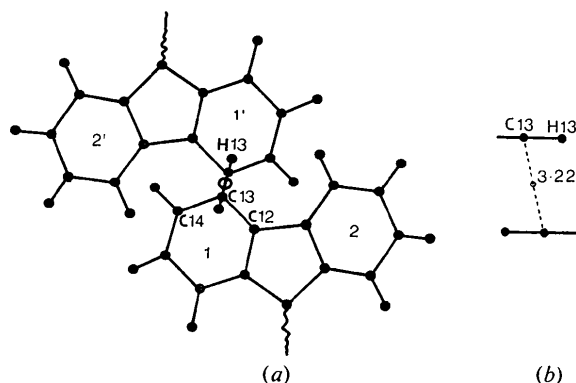


Fig. 3. (a) Projection on the plane of ring 1 of rings 1 and 2 and their inversions at  $x=y=0.5$  and  $z=0$ . (b) Cross section through C(13), H(13), C(13'), H(13') perpendicular to ring 1.

Table 4. *The valence angles (°) of BBM*

Corresponding angles of the four rings are given in rows with their mean values of which the experimental standard deviation is given in parentheses. The e.s.d.'s of the individual angles are 0.4–0.5° for C–C–C angles and 3–4° for C–C–H angles.

	Mean value	Ring 1	Ring 2	Ring 3	Ring 4
		<i>i</i> =1 <i>j</i> =2 <i>k</i> =3	<i>i</i> =2 <i>j</i> =1 <i>k</i> =4	<i>i</i> =3 <i>j</i> =4 <i>k</i> =2	<i>i</i> =4 <i>j</i> =3 <i>k</i> =1
C( <i>i</i> 1)–C(1)–C( <i>j</i> 1)	101.1 (1)	101.2		100.9	
C( <i>i</i> 1)–C(1)–C( <i>k</i> 1)	113.8 (6)	113.7	115.3	114.1	112.2
C(1)–C( <i>i</i> 1)–C( <i>i</i> 2)	110.9 (1)	110.7	111.1	110.6	111.1
C(1)–C( <i>i</i> 1)–C( <i>i</i> 6)	127.9 (2)	127.8	128.6	128.0	127.3
C( <i>i</i> 2)–C( <i>i</i> 1)–C( <i>i</i> 6)	121.2 (3)	121.5	120.3	121.3	121.5
C( <i>i</i> 1)–C( <i>i</i> 2)–C( <i>i</i> 3)	120.6 (1)	120.9	120.8	120.2	120.4
C( <i>i</i> 1)–C( <i>i</i> 2)–C( <i>j</i> 2)	108.6 (3)	109.1	108.0	109.0	108.2
C( <i>i</i> 3)–C( <i>i</i> 2)–C( <i>j</i> 2)	130.8 (3)	130.0	131.2	130.7	131.4
C( <i>i</i> 2)–C( <i>i</i> 3)–C( <i>i</i> 4)	118.1 (2)	117.6	118.5	118.1	118.3
C( <i>i</i> 2)–C( <i>i</i> 3)–H( <i>i</i> 3)	121 (1)	123	120	122	120
C( <i>i</i> 4)–C( <i>i</i> 3)–H( <i>i</i> 3)	120 (1)	119	121	119	122
C( <i>i</i> 3)–C( <i>i</i> 4)–C( <i>i</i> 5)	121.2 (2)	121.5	120.8	120.9	121.7
C( <i>i</i> 3)–C( <i>i</i> 4)–H( <i>i</i> 4)	120 (2)	118	117	118	127
C( <i>i</i> 5)–C( <i>i</i> 4)–H( <i>i</i> 4)	119 (2)	121	122	121	111
C( <i>i</i> 4)–C( <i>i</i> 5)–C( <i>i</i> 6)	121.2 (1)	121.4	120.9	121.2	121.1
C( <i>i</i> 4)–C( <i>i</i> 5)–H( <i>i</i> 5)	125 (3)	126	120	122	132
C( <i>i</i> 6)–C( <i>i</i> 5)–H( <i>i</i> 5)	114 (3)	113	120	117	107
C( <i>i</i> 1)–C( <i>i</i> 6)–C( <i>i</i> 5)	117.8 (4)	117.1	118.8	118.2	117.1
C( <i>i</i> 1)–C( <i>i</i> 6)–H( <i>i</i> 6)	120 (1)	121	121	121	118
C( <i>i</i> 5)–C( <i>i</i> 6)–H( <i>i</i> 6)	122 (1)	122	120	121	125

#### Planarity

The individual rings (planes 2, 3, 5 and 6 in Table 5) are planar. However from the distances of the atoms to these planes it is evident that both biphenyl groups of the molecule deviate significantly from planarity. The angle between rings 1 and 2 is 1.2° and that between rings 3 and 4 is 4.2°. These effects can be attributed to the packing and cause the symmetry  $\bar{4}m2$  of the free molecule to disappear in the crystal.

#### Crystal structure

The packing of the molecules is illustrated in Figs. 2 and 3 and the short intermolecular contacts are given in Table 6. The most striking feature is the occurrence of very short intermolecular C–C distances (3.22, 3.35 and 3.45 Å) approximately perpendicular to the planes of the benzene rings, which normally are found only in charge transfer complexes. The shortest occurs between ring 1 and its inversion at  $\frac{1}{2}, \frac{1}{2}, 0$  [see Fig. 3(a)]. The other two occur between ring 4 and its inversion at 0, 0, 0, which are similarly situated as is evident from Fig. 2. Thus the usual van der Waals radius of 1.8 Å for carbon definitely does not apply to the C...C distance in the direction indicated in Fig. 3(b).

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Table 5. Planes through the benzene rings of *BBM*

The coordinates  $x$ ,  $y$  and  $z$  in the expressions for the planes are orthogonal and given in Å (Cruickshank, 1961).

Plane number	Carbon atoms defining the plane			Equation of the plane		
	1	C(1) + ring 1 + ring 2			$0.310x - 0.385y - 0.870z + 1.980 = 0$	
2	C(1) + ring 1			$0.304x - 0.383y - 0.873z + 1.990 = 0$		
3	C(1) + ring 2			$0.316x - 0.396y - 0.862z + 1.939 = 0$		
4	C(1) + ring 3 + ring 4			$0.613x - 0.644y + 0.458z - 1.615 = 0$		
5	C(1) + ring 3			$0.615x - 0.668y + 0.419z - 1.412 = 0$		
6	C(1) + ring 4			$0.602x - 0.636y + 0.484z - 1.614 = 0$		

	Distance in Å to plane*			Distance in Å to plane*			
	1	2	3	4	5	6	
C(1)	<b>0.02</b>	<b>0.01</b>	<b>-0.01</b>	C(1)	<b>-0.06</b>	<b>0.00</b>	<b>-0.01</b>
C(11)	<b>-0.01</b>	<b>-0.01</b>	<b>-0.05</b>	C(31)	<b>-0.04</b>	<b>-0.01</b>	<b>0.05</b>
C(12)	<b>0.01</b>	<b>0.00</b>	<b>-0.04</b>	C(32)	<b>-0.05</b>	<b>0.01</b>	<b>0.03</b>
C(13)	<b>0.00</b>	<b>0.00</b>	<b>-0.06</b>	C(33)	<b>-0.04</b>	<b>0.00</b>	<b>0.07</b>
C(14)	<b>-0.01</b>	<b>0.00</b>	<b>-0.08</b>	C(34)	<b>0.02</b>	<b>-0.01</b>	<b>0.16</b>
C(15)	<b>-0.01</b>	<b>0.00</b>	<b>-0.09</b>	C(35)	<b>0.08</b>	<b>0.01</b>	<b>0.23</b>
C(16)	<b>-0.01</b>	<b>0.00</b>	<b>-0.08</b>	C(36)	<b>0.04</b>	<b>0.00</b>	<b>0.16</b>
C(21)	<b>0.02</b>	<b>0.00</b>	<b>0.01</b>	C(41)	<b>-0.01</b>	<b>0.12</b>	<b>0.01</b>
C(22)	<b>0.02</b>	<b>0.01</b>	<b>0.00</b>	C(42)	<b>-0.02</b>	<b>0.10</b>	<b>0.01</b>
C(23)	<b>0.01</b>	<b>-0.01</b>	<b>0.00</b>	C(43)	<b>0.00</b>	<b>0.18</b>	<b>0.00</b>
C(24)	<b>-0.01</b>	<b>-0.05</b>	<b>0.00</b>	C(44)	<b>0.02</b>	<b>0.26</b>	<b>-0.01</b>
C(25)	<b>-0.03</b>	<b>-0.06</b>	<b>-0.01</b>	C(45)	<b>0.05</b>	<b>0.28</b>	<b>0.00</b>
C(26)	<b>-0.01</b>	<b>-0.03</b>	<b>0.01</b>	C(46)	<b>0.04</b>	<b>0.22</b>	<b>0.01</b>

\*distances in bold type refer to atoms defining the planes.

Interplanar angles in degrees between plane *A* and *B*

plane A → plane B ↓	1	2	3	4	5	6
1	-	0.4	0.9	87.7	85.2	89.4
2		-	1.2	88.1	85.6	89.8
3			-	86.9	84.3	88.5
4				-	2.6	1.7
5					-	4.2
6						-

Table 6. Short intermolecular atom-atom distances

Hydrogen-hydrogen distances are given up to 2.50 Å, hydrogen-carbon up to 3.0 and carbon-carbon up to 3.7 Å.

First atom	Second atom	Equivalent position of the second atom	Distance
C(12)	H(26)	$1-x \quad \frac{1}{2}+y \quad \frac{1}{2}-z$	2.92 Å
C(15)	H(34)	$-x \quad \frac{1}{2}+y \quad \frac{1}{2}-z$	2.91
C(22)	H(26)	$1-x \quad \frac{1}{2}+y \quad \frac{1}{2}-z$	2.90
C(25)	H(45)	$1-x \quad -y \quad -z$	2.74
C(26)	H(45)	$1-x \quad -y \quad -z$	2.71
C(33)	H(23)	$1-x \quad -\frac{1}{2}-y \quad \frac{1}{2}-z$	2.97
C(34)	H(23)	$1-x \quad -\frac{1}{2}-y \quad \frac{1}{2}-z$	2.96
C(34)	H(24)	$-1+x \quad y \quad z$	2.97
C(35)	H(43)	$-x \quad \frac{1}{2}-y \quad \frac{1}{2}-z$	2.99
C(44)	H(14)	$x \quad -1+y \quad z$	2.81
H(15)	H(16)	$-x \quad 1-y \quad -z$	2.46
H(35)	H(43)	$-x \quad \frac{1}{2}+y \quad \frac{1}{2}-z$	2.34
C(12)	C(13)	$1-x \quad 1-y \quad -z$	3.65
C(13)	C(14)	$1-x \quad 1-y \quad -z$	3.63
C(13)	C(13)	$1-x \quad 1-y \quad -z$	3.22
C(43)	C(43)	$-x \quad -y \quad -z$	3.35
C(43)	C(44)	$-x \quad -y \quad -z$	3.45

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