

## The Crystal and Molecular Structure of Hexacyclo[10,3,1,0<sup>2,10</sup>,0<sup>3,7</sup>,0<sup>6,15</sup>,0<sup>9,14</sup>]hexadecane, an Ethano-Bridged Diamantane

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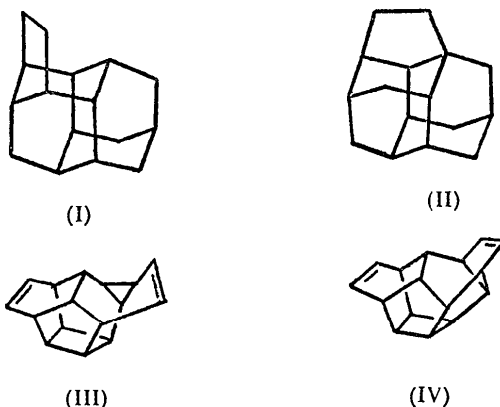
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The structure of a hydrocarbon, m.p. 110–115°C, obtained from two different cyclooctatetraene dimers by hydrogenation followed by aluminum halide rearrangement, has been determined to be an ethano-diamantane, hexacyclo[10,3,1,0<sup>2,10</sup>,0<sup>3,7</sup>,0<sup>6,15</sup>,0<sup>9,14</sup>]hexadecane. The crystals are monoclinic with unit-cell dimensions:  $a = 9.151 \pm 0.001$ ,  $b = 6.510 \pm 0.001$ ,  $c = 19.733 \pm 0.003$  Å and  $\beta = 98.62 \pm 0.02^\circ$ . The space group is  $P2_1/c$  with four molecules in the unit cell. The structure was solved by a straightforward application of the symbolic addition procedure and refined by full-matrix least-squares methods with anisotropic thermal parameters for the carbon atoms and isotropic thermal parameters for the hydrogen atoms. The final  $R$  value is 0.044 for 1615 observed reflections. The average standard deviation in the C–C bond distances is 0.002 Å and that in the C–H bond is 0.02 Å. The C–C bond distances lie in the range 1.516–1.552 Å (mean 1.534 Å), and the bond angles range from 99.2 to 114.5° (mean 109.1°). The molecule has seven six-membered rings; the mean torsion angle in all of them is greater than the value of 56° found in cyclohexane itself. The cyclopentane ring is in the envelope conformation with the puckered atom deviating 0.4 Å from the plane of the remaining atoms.

### Introduction

The ethano-bridged diamantane molecules I and II are prepared by aluminum halide rearrangement of two different isomeric polycyclic hydrocarbons III and IV. Isomer I (about 15%) had a melting point of 110–111.5°, while II (about 85%) melted between 206.5–208°. The lower melting isomer formed suitable crystals and



the X-ray structure analysis of this compound was initiated to determine which of the two possible structures (I or II) was actually correct. A preliminary communication on this investigation including details of the chemical reactions has already been published (Rao, Sundaralingam, Osawa, Wiskott & Schleyer, 1970).

The crystal structures of four other cage hydrocarbons, congressane (Karle & Karle, 1965), adamantane (Nordman & Schmitkors, 1965), diadamantane (Allden, Kraut & Taylor, 1968) and tetramantane (Schleyer, Osawa & Drew, 1968) have been determined

to date. In the former two cases the molecules occupy a special position in the lattice and thus are constrained to the corresponding symmetry. In diadamantane the two halves of the molecule are related by an inversion center.

### Experimental

Crystals of the compound were supplied by Professor P. von R. Schleyer of Princeton University. A crystal of dimensions 0.15 × 0.2 × 0.5 mm was chosen for the analysis and was sealed inside a Lindemann capillary tube to minimize sublimation. Preliminary oscillation and Weissenberg photographs showed the crystal to be monoclinic with space group  $P2_1/c$ . The cell constants (Table 1) were determined using reflections with medium  $2\theta$  angle on a Picker four-circle diffractometer and Ni-filtered Cu radiation. Intensity data were collected by the  $\theta$ - $2\theta$  scan method at a scan speed of 2° min<sup>-1</sup> on the Picker four-circle diffractometer. The crystal was mounted with the  $b$  axis coincident with the  $\varphi$  axis of the goniostat. 2080 reflections with  $2\theta \leq 134^\circ$  were measured. Using the criterion that  $I > 1.5\sigma(I)$ , 1615 reflections were considered observed, and were corrected for the usual Lorentz and polarization factors.

Table 1. *Crystal data*

Stoichiometry	C <sub>16</sub> H <sub>22</sub>
Cell dimensions	$a = 9.151 \pm 0.001$ Å $b = 6.510 \pm 0.001$ $c = 19.733 \pm 0.003$ $\beta = 98.62 \pm 0.02^\circ$
Systematic absences	$0k0$ , $k = 2n + 1$ ; $h0l$ , $l = 2n + 1$
Space group	$P2_1/c$
$Z$	4
Observed density	1.220 g.cm <sup>-3</sup>
Calculated density	1.228 g.cm <sup>-3</sup>
$\mu$ for Cu $K\alpha$	9.2 cm <sup>-1</sup>

### Structure analysis and refinement

The intensity distribution showed characteristics of hypercentering. The structure was solved by a straightforward application of the symbolic addition procedure (Karle & Karle, 1966). Two hundred sixty-eight reflections with  $E > 1.4$  were used for the sign determination. The three origin-determining signs and three symbolic phases used initially are given in Table 2. The signs were generated by a program written by Long (1965). The most probable solution was found to be correct, and the  $E$  map calculated with these signs showed all the carbon atoms in the structure.

Table 2. Reflections used to assign the origin phases and initiate the process of sign determination

$h$	$k$	$l$	$E$	Sign
-4	3	10	5.21	+
3	3	10	4.11	+
3	1	13	4.00	+
				} origin
0	3	6	3.84	$a$
0	1	2	3.44	$b$
-4	1	6	3.26	$c$

Three cycles of isotropic full-matrix least-squares (Busing, Martin & Levy, 1962) reduced the  $R$  value to 0.09. A difference electron-density map calculated at this stage revealed the positions of all the hydrogen atoms. The refinement was continued, with anisotropic thermal parameters for the heavy atoms and isotropic thermal parameters for the hydrogen atoms, until the shifts in the parameters were less than 10% of the estimated standard deviations. The final  $R$  value was 0.044 for the observed reflections and 0.064 for all the reflections including the unobserved ones. The scattering factors of carbon were taken from *International Tables for X-ray Crystallography* (1962), while those of hydrogen were taken from Stewart, Davidson & Simpson (1965). The weighting scheme was based on the

counting statistics with an electronic instability of 2% (Stout & Jensen, 1968).

The positional and thermal parameters of the heavy atoms are listed in Table 3; those of hydrogen atoms are listed in Table 4. An *ORTEP* plot (Johnson, 1965) of the molecule in stereo is presented in Fig. 1. The anisotropic thermal ellipsoids have essentially similar semi-axes. The thermal parameters of the hydrogen atoms are very reasonable, and those of the hydrogen atoms H(4') and H(5') in the ethano-bridge are the smallest, because they are less free to vibrate than the others. The observed and calculated structure factors are given in Table 5, where the low-angle reflections suspected of secondary extinction are marked with an 'E' and were given zero weight during refinement. The unobserved reflections are marked with an asterisk.

Table 4. Coordinates and isotropic thermal parameters of the hydrogen atoms

Coordinates are  $\times 10^4$  and standard deviations refer to the least significant digits

	$x$	$y$	$z$	$B(\text{\AA}^2)$
H(1)	6099 (16)	1855 (22)	2799 (7)	1.1
H(2)	6643 (17)	5364 (25)	3099 (8)	1.9
H(3)	5236 (17)	5583 (24)	4022 (8)	2.3
H(4)	3154 (25)	3712 (36)	3623 (10)	6.6
H(4')	4059 (18)	3513 (26)	2913 (8)	2.8
H(5)	3487 (23)	254 (33)	3814 (10)	6.1
H(5')	4362 (17)	-56 (25)	3125 (8)	2.2
H(6)	5843 (20)	-471 (30)	4388 (9)	4.3
H(7)	4926 (19)	2710 (26)	4758 (8)	3.5
H(8)	7428 (20)	2039 (28)	5332 (9)	4.2
H(8')	7174 (17)	4510 (25)	5137 (8)	2.2
H(9)	9494 (17)	3333 (23)	4808 (7)	2.1
H(10)	8107 (19)	6023 (27)	4178 (9)	3.5
H(11)	9425 (18)	5770 (25)	3187 (8)	2.3
H(11')	10482 (21)	5056 (31)	3907 (10)	4.8
H(12)	10580 (21)	2572 (28)	2980 (9)	4.3
H(13)	10797 (21)	1237 (30)	4159 (9)	4.6
H(13')	9982 (19)	-402 (27)	3577 (9)	3.5
H(14)	8670 (18)	-116 (25)	4572 (8)	2.8
H(15)	7207 (19)	-797 (28)	3478 (8)	3.5
H(16)	8164 (17)	3280 (28)	2980 (9)	4.3
H(16')	8390 (17)	770 (25)	2565 (8)	2.2

Table 3. Coordinates and anisotropic thermal parameters of the carbon atoms

All numbers are multiplied by  $10^5$  and standard deviations refer to the least significant digits.

	$x$	$y$	$z$	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
C(1)	69871 (16)	22048 (25)	31272 (7)	878 (19)	2015 (44)	177 (4)	-70 (24)	36 (7)	-46 (11)
C(2)	68089 (17)	42724 (24)	34778 (7)	1042 (21)	1689 (41)	203 (5)	81 (24)	57 (8)	79 (11)
C(3)	54690 (18)	41977 (26)	38682 (8)	1044 (23)	2375 (49)	237 (5)	323 (28)	93 (9)	-6 (13)
C(4)	41050 (19)	31780 (33)	34450 (9)	914 (23)	3975 (69)	304 (6)	160 (32)	67 (9)	3 (17)
C(5)	43142 (20)	8429 (33)	35845 (9)	1083 (26)	3873 (69)	325 (6)	-688 (35)	118 (10)	-21 (17)
C(6)	57782 (19)	6709 (28)	40780 (8)	1269 (26)	2425 (53)	261 (5)	363 (29)	100 (10)	128 (13)
C(7)	58016 (18)	27029 (29)	44696 (8)	1131 (23)	3059 (58)	201 (4)	-47 (30)	154 (8)	-2 (13)
C(8)	72375 (20)	31225 (29)	49418 (8)	1466 (27)	3119 (59)	132 (4)	17 (33)	62 (9)	-37 (13)
C(9)	84951 (17)	30709 (26)	45220 (8)	1083 (22)	2183 (47)	198 (4)	-37 (26)	-42 (8)	-41 (12)
C(10)	82583 (18)	47255 (24)	39606 (8)	1124 (23)	1665 (43)	229 (5)	-148 (24)	54 (9)	-73 (11)
C(11)	95799 (19)	47326 (28)	35659 (9)	1151 (25)	2660 (55)	299 (6)	-470 (30)	101 (10)	-16 (15)
C(12)	97254 (17)	26211 (30)	32469 (9)	890 (21)	3183 (59)	277 (5)	-99 (29)	131 (9)	-120 (15)
C(13)	99124 (19)	9471 (29)	37925 (9)	1089 (25)	2728 (56)	342 (6)	378 (30)	20 (10)	-95 (15)
C(14)	85850 (18)	9655 (25)	41818 (8)	1113 (23)	1872 (44)	242 (5)	175 (26)	-30 (9)	59 (12)
C(15)	71276 (17)	5698 (24)	36935 (8)	1126 (23)	1572 (41)	231 (4)	-98 (25)	41 (8)	-29 (11)
C(16)	83149 (18)	21918 (28)	27440 (8)	1105 (23)	2909 (55)	221 (5)	-56 (30)	129 (8)	-138 (13)

Discussion

The bond lengths and angles involving the carbon atoms are given in Fig. 2. The average standard deviation in the C-C distances is 0.002 Å (range 0.0015 to 0.0025 Å) and that in the C-C-C angle is 0.13° (range 0.11 to 0.15°). The molecule may be regarded as having

a mirror plane containing atoms 1, 16, 12, 9, 8, 7 and the midpoint of the 4-5 bond. The C-C distances listed in Table 6 have been classified into the different bond types. A bond type for a given bond is specified by two numbers representing the number of C-C bonds in which the terminal carbon atoms participate. Except for the bonds involved in the strained bridge, the bonds

Table 5. Observed and calculated structure factors ( $\times 10$ )

h	k	l	obs	calc	h	k	l	obs	calc	h	k	l	obs	calc
1	0	0	1.00	1.00	1	1	0	0.20	0.20	1	1	1	0.15	0.15
2	0	0	0.40	0.40	2	0	0	0.10	0.10	2	1	0	0.08	0.08
3	0	0	0.15	0.15	3	0	0	0.05	0.05	3	1	0	0.04	0.04
4	0	0	0.08	0.08	4	0	0	0.03	0.03	4	1	0	0.02	0.02
5	0	0	0.04	0.04	5	0	0	0.02	0.02	5	1	0	0.01	0.01
6	0	0	0.02	0.02	6	0	0	0.01	0.01	6	1	0	0.00	0.00
7	0	0	0.01	0.01	7	0	0	0.00	0.00	7	1	0	0.00	0.00
8	0	0	0.00	0.00	8	0	0	0.00	0.00	8	1	0	0.00	0.00
9	0	0	0.00	0.00	9	0	0	0.00	0.00	9	1	0	0.00	0.00
10	0	0	0.00	0.00	10	0	0	0.00	0.00	10	1	0	0.00	0.00
11	0	0	0.00	0.00	11	0	0	0.00	0.00	11	1	0	0.00	0.00
12	0	0	0.00	0.00	12	0	0	0.00	0.00	12	1	0	0.00	0.00
13	0	0	0.00	0.00	13	0	0	0.00	0.00	13	1	0	0.00	0.00
14	0	0	0.00	0.00	14	0	0	0.00	0.00	14	1	0	0.00	0.00
15	0	0	0.00	0.00	15	0	0	0.00	0.00	15	1	0	0.00	0.00
16	0	0	0.00	0.00	16	0	0	0.00	0.00	16	1	0	0.00	0.00
17	0	0	0.00	0.00	17	0	0	0.00	0.00	17	1	0	0.00	0.00
18	0	0	0.00	0.00	18	0	0	0.00	0.00	18	1	0	0.00	0.00
19	0	0	0.00	0.00	19	0	0	0.00	0.00	19	1	0	0.00	0.00
20	0	0	0.00	0.00	20	0	0	0.00	0.00	20	1	0	0.00	0.00
21	0	0	0.00	0.00	21	0	0	0.00	0.00	21	1	0	0.00	0.00
22	0	0	0.00	0.00	22	0	0	0.00	0.00	22	1	0	0.00	0.00
23	0	0	0.00	0.00	23	0	0	0.00	0.00	23	1	0	0.00	0.00
24	0	0	0.00	0.00	24	0	0	0.00	0.00	24	1	0	0.00	0.00
25	0	0	0.00	0.00	25	0	0	0.00	0.00	25	1	0	0.00	0.00
26	0	0	0.00	0.00	26	0	0	0.00	0.00	26	1	0	0.00	0.00
27	0	0	0.00	0.00	27	0	0	0.00	0.00	27	1	0	0.00	0.00
28	0	0	0.00	0.00	28	0	0	0.00	0.00	28	1	0	0.00	0.00
29	0	0	0.00	0.00	29	0	0	0.00	0.00	29	1	0	0.00	0.00
30	0	0	0.00	0.00	30	0	0	0.00	0.00	30	1	0	0.00	0.00
31	0	0	0.00	0.00	31	0	0	0.00	0.00	31	1	0	0.00	0.00
32	0	0	0.00	0.00	32	0	0	0.00	0.00	32	1	0	0.00	0.00
33	0	0	0.00	0.00	33	0	0	0.00	0.00	33	1	0	0.00	0.00
34	0	0	0.00	0.00	34	0	0	0.00	0.00	34	1	0	0.00	0.00
35	0	0	0.00	0.00	35	0	0	0.00	0.00	35	1	0	0.00	0.00
36	0	0	0.00	0.00	36	0	0	0.00	0.00	36	1	0	0.00	0.00
37	0	0	0.00	0.00	37	0	0	0.00	0.00	37	1	0	0.00	0.00
38	0	0	0.00	0.00	38	0	0	0.00	0.00	38	1	0	0.00	0.00
39	0	0	0.00	0.00	39	0	0	0.00	0.00	39	1	0	0.00	0.00
40	0	0	0.00	0.00	40	0	0	0.00	0.00	40	1	0	0.00	0.00
41	0	0	0.00	0.00	41	0	0	0.00	0.00	41	1	0	0.00	0.00
42	0	0	0.00	0.00	42	0	0	0.00	0.00	42	1	0	0.00	0.00
43	0	0	0.00	0.00	43	0	0	0.00	0.00	43	1	0	0.00	0.00
44	0	0	0.00	0.00	44	0	0	0.00	0.00	44	1	0	0.00	0.00
45	0	0	0.00	0.00	45	0	0	0.00	0.00	45	1	0	0.00	0.00
46	0	0	0.00	0.00	46	0	0	0.00	0.00	46	1	0	0.00	0.00
47	0	0	0.00	0.00	47	0	0	0.00	0.00	47	1	0	0.00	0.00
48	0	0	0.00	0.00	48	0	0	0.00	0.00	48	1	0	0.00	0.00
49	0	0	0.00	0.00	49	0	0	0.00	0.00	49	1	0	0.00	0.00
50	0	0	0.00	0.00	50	0	0	0.00	0.00	50	1	0	0.00	0.00
51	0	0	0.00	0.00	51	0	0	0.00	0.00	51	1	0	0.00	0.00
52	0	0	0.00	0.00	52	0	0	0.00	0.00	52	1	0	0.00	0.00
53	0	0	0.00	0.00	53	0	0	0.00	0.00	53	1	0	0.00	0.00
54	0	0	0.00	0.00	54	0	0	0.00	0.00	54	1	0	0.00	0.00
55	0	0	0.00	0.00	55	0	0	0.00	0.00	55	1	0	0.00	0.00
56	0	0	0.00	0.00	56	0	0	0.00	0.00	56	1	0	0.00	0.00
57	0	0	0.00	0.00	57	0	0	0.00	0.00	57	1	0	0.00	0.00
58	0	0	0.00	0.00	58	0	0	0.00	0.00	58	1	0	0.00	0.00
59	0	0	0.00	0.00	59	0	0	0.00	0.00	59	1	0	0.00	0.00
60	0	0	0.00	0.00	60	0	0	0.00	0.00	60	1	0	0.00	0.00

fall into two bond types: (2)–(3) and (3)–(3). The following trend, though not very pronounced, is apparent in the C–C bond distance (3)–(3) > (2)–(3).

Table 6. Classification of bond lengths in terms of bond type

	Bond type†	Length (Å)	Mean C–C distance
C(11)–C(12)	(2)–(3)	1.526‡	1.527
C(12)–C(13)		1.523	
C(12)–C(16)		1.531	
C(1)–C(16)	(2)–(3)	1.524	1.525
C(7)–C(9)		1.517	
C(8)–C(9)		1.516	
C(10)–C(11)		1.534	
C(13)–C(14)		1.532	
C(3)–C(7)	(3)–(3)	1.530	1.533
C(6)–C(7)		1.531	
C(9)–C(10)		1.537	
C(9)–C(14)		1.534	
C(1)–C(2)	(3)–(3)	1.533	1.539
C(1)–C(15)		1.535	
C(2)–C(3)		1.544	
C(2)–C(10)		1.541	
C(6)–C(15)		1.545	
C(4)–C(15)		1.545	
*C(4)–C(5)	(2)–(2)	1.552	1.552
*C(3)–C(4)	(2)–(3)	1.544	1.541
*C(5)–C(6)		1.538	

\* Bonds in the bridge.

† Denotes nearest neighbor.

‡ The average standard deviation in the bond lengths is 0.002 Å.

The longest C–C distance of 1.552 Å is associated with the bridge bond C(4)–C(5) [bond type (2)–(2)]. The marked lengthening in this bond is primarily due to the interaction between the eclipsed hydrogen atoms on C(4) and C(5). Similarly, the lengthening in the C(4)–C(3) and C(5)–C(6) bonds [bond type (2)–(3)] is due to the partially staggered bonds around them.

The C–C–C bond angles vary from 99.9 to 114.5°

with a mean of 109.1°. The largest and smallest of these angles are associated with the atom C(7) at the bridge-head position of the ethano-bridged adamantane moiety. All of the endocyclic angles in the 5-membered ring are significantly smaller than the ideal tetrahedral value.

The C–H distances range from 0.96 to 1.09 Å with a mean of  $1.02 \pm 0.02$  Å. The C–C–H and H–C–H angles have mean values of  $109.7 \pm 0.8$  and  $111 \pm 1^\circ$  respectively.

The molecule can be thought of as being composed of seven cyclohexane rings and one cyclopentane ring. The mean values of the endocyclic torsional angles of the cyclohexane ring systems together with the mean bond angles, are listed in Table 7. The mean torsional angles in all seven rings are greater than the mean value of  $56^\circ$  in cyclohexane itself (Altona & Sundaralingam, 1970). The greatest- and least-puckered cyclohexane rings are involved in the fusion with the ethano bridge. It is also seen that the mean bond angle decreases from the ideal tetrahedral value with increasing puckering of the ring.

Table 7. The endocyclic torsional angles and bond angles in the various cyclohexane ring systems\*

No.	Atoms	Torsion angle Range	Mean	Mean bond angle
1	C(1), C(2), C(10), C(11), C(12), C(16)	56.7–61.5°	59.4 (2)°	109.7 (1)°
2	C(9), C(10), C(11), C(12), C(13), C(14)	58.0–61.7	59.8 (2)	109.5 (1)
3	C(1), C(15), C(14), C(13), C(12), C(16)	56.8–60.7	59.1 (2)	109.8 (1)
4	C(1), C(2), C(10), C(9), C(14), C(15)	59.7–65.8	62.9 (2)	108.2 (1)
5	C(1), C(2), C(3), C(6), C(7), C(15)	56.1–69.3	64.2 (2)	107.5 (1)
6	C(2), C(3), C(7), C(8), C(9), (10)	52.7–61.5	57.2 (2)	110.5 (1)
7	C(6), C(7), C(8), C(9), C(14), C(15)	53.2–61.3	57.1 (2)	110.6 (1)

\* The six-membered rings 1, 2 and 3 form the top adamantane system (Fig. 3), while the rings 5, 6, and 7 form the ethano-bridged adamantane ring. Ring 4 is common to both adamantane rings.

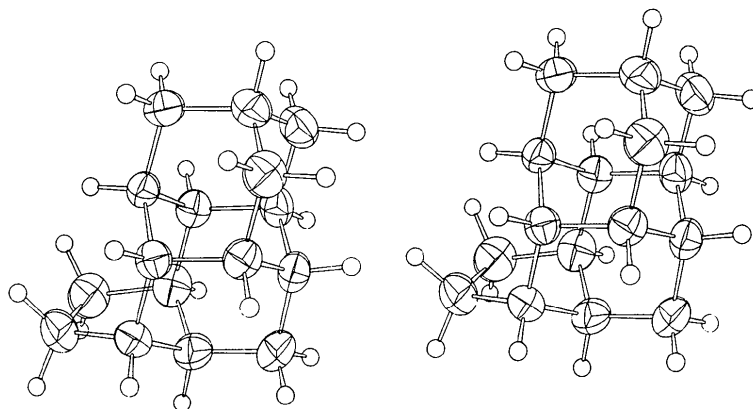


Fig. 1. A stereoscopic diagram of the molecule.

## AN ETHANO-BRIDGED DIAMANTANE

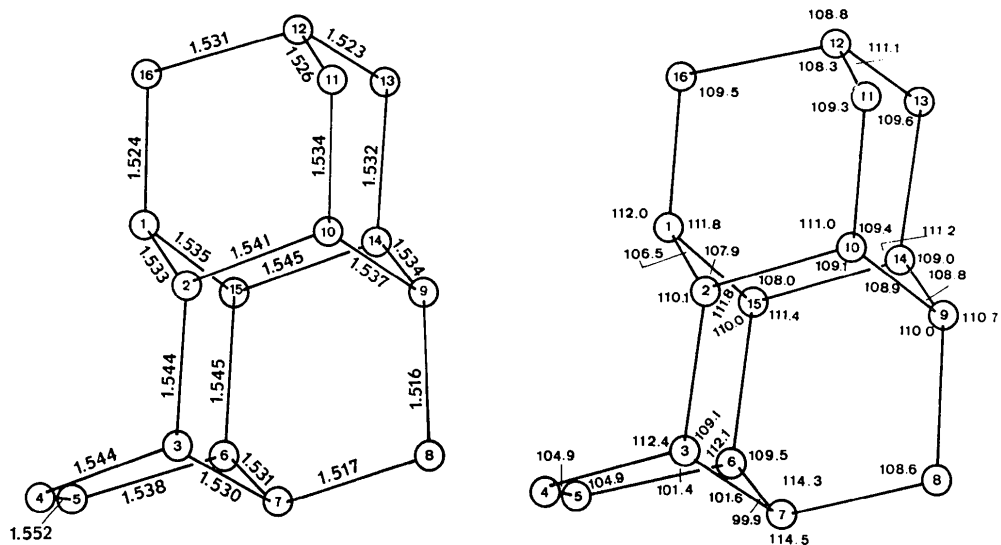


Fig. 2. Bond lengths and bond angles in the carbon skeleton. The average standard deviation in the bond lengths is 0.002 Å and that in the bond angles is 0.13°.

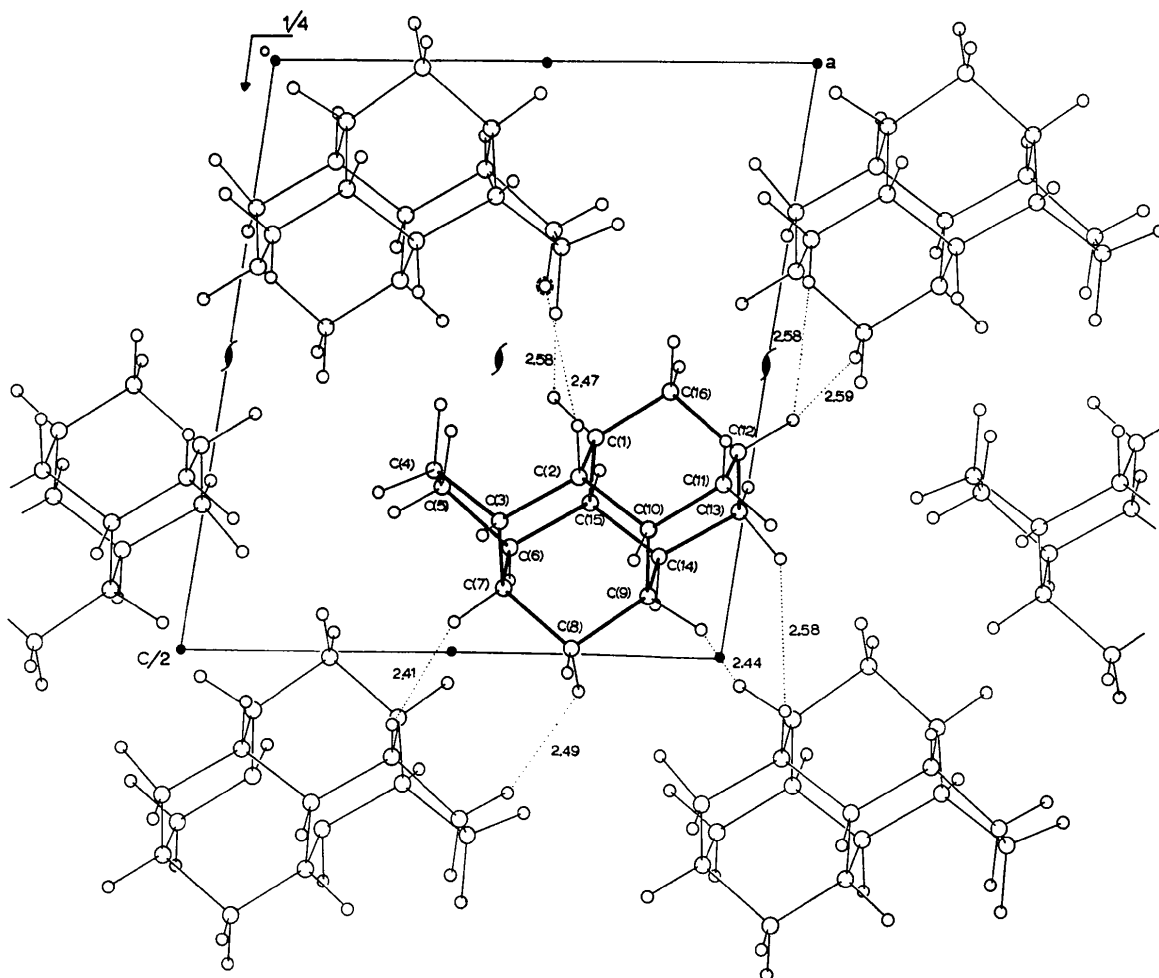


Fig. 3. Packing diagram of the structure viewed down the *b* axis.

The cyclopentane ring is in the envelope form rather than the common half-chair form. The carbon atoms 3, 4, 5 and 6 lie on a plane. The methylene groups on C(4) and C(5) are eclipsed, with a twist of only  $1^\circ$  around the C(4)–C(5) bond. The smallest valence angle is at the puckered carbon atom C(7).

The carbon skeleton is not significantly twisted; the twists around the pseudo bonds C(2)–C(15), C(10)–C(14) and C(3)–C(6) are less than  $0.1^\circ$ . The H–C–C–H torsional angles around the C–C bonds in the cyclohexane ring systems vary from  $52$  to  $70^\circ$  with a mean of  $59 \pm 2^\circ$ . The smallest angle is around the C(2)–C(3) bond and the largest one is around the C(9)–C(10) bond. The H–C–C–H torsion angles around the C(3)–C(4) and C(5)–C(6) bonds in the cyclopentane ring are ( $-31, 91^\circ$ ) and ( $30, -87^\circ$ ) and are semi-staggered.

The packing diagram of the structure as viewed down the  $b$  axis is shown in Fig. 3. There are no unusually short contacts in the structure, and the H---H distances less than  $2.6 \text{ \AA}$  are shown in the Figure.

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## Die Strukturen des Moleküls und des zweifach negativ geladenen Anions der *trans*-Cyclohexandicarbonsäure(1,4)

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The structure of a potassium salt of *trans*-cyclohexane-1,4-dicarboxylic acid, chemical formula  $2\text{C}_8\text{H}_{11}\text{O}_4\text{K} \cdot \text{C}_8\text{H}_{12}\text{O}_4$  or  $\text{C}_8\text{H}_{10}\text{O}_4\text{K}_2 \cdot 2\text{C}_8\text{H}_{12}\text{O}_4$ , was solved by X-ray analysis. Investigations of the bond lengths of the carboxylic groups led to the conclusion that the correct formula is  $\text{C}_8\text{H}_{10}\text{O}_4\text{K}_2 \cdot 2\text{C}_8\text{H}_{12}\text{O}_4$ .

Wie wir bereits mitgeteilt haben (Luger, Plieth & Ruban, 1970) gelang uns im Rahmen einer Untersuchungsreihe an Cyclohexanderivaten die Darstellung des Mono-Kalium-sesqui[Cyclohexandicarbonsäure(1,4)]-salzes. Mit den vorläufigen Ergebnissen einer röntgenographischen Strukturbestimmung konnten wir zeigen, dass die Substanz in der Raumgruppe  $P\bar{1}$  mit zwei  $\text{K}^+$ -Ionen und drei Säure- bzw. Säurerestmolekülen kristallisiert. Kristallographische Daten sind in Tabelle 1 enthalten.

Dabei befindet sich nicht nur der Schwerpunkt eines Cyclohexanringes in einem Symmetriezentrum, sondern alle drei Moleküle sind um die speziellen Lagen  $\frac{1}{2}, 0, \frac{1}{2}$ ;  $0, 0, \frac{1}{2}$  bzw.  $\frac{1}{2}, \frac{1}{2}, 0$  angeordnet.

Im Rahmen dieser Arbeit sollen die Ergebnisse einer

weiteren Verfeinerung der Struktur mitgeteilt werden. Es konnten die Parameter sämtlicher Wasserstoffatome bestimmt werden und aus den Bindungslängen an den Carboxylgruppen die Entscheidung zugunsten einer der beiden möglichen chemischen Formeln getroffen werden. Das nach der Schweratommethode über die Kalium-Parameter bestimmte Strukturmodell wurde mit anisotropen Temperaturfaktoren für alle Atome durch das least-squares Programm *ORFLS* des Programmsystems *X-ray 63* (1963) bis zu einem  $R$ -Wert von 8,3% verfeinert. Einer in diesem Stadium berechneten Differenzsynthese konnten sämtliche Wasserstoffatomlagen entnommen werden. Weitere Verfeinerungen, die bei den Wasserstoffatomen jedoch isotrop durchgeführt wurden, konvergierten bei einem  $R$ -Wert von 6,3%.