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The Crystal and Molecular Structure of Bromdihydroacronycine

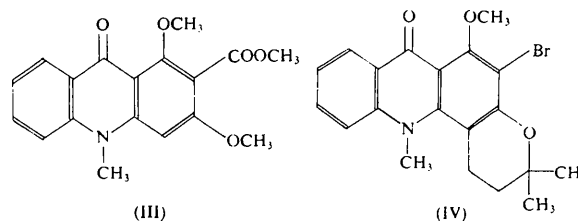
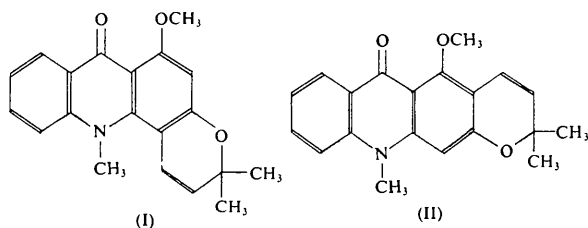
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Bromdihydroacronycine (1-methoxy-2-bromo-2',2',10-trimethyldihydropyranof[5',6'-4,3]acrid-9-one, $C_{20}H_{20}NO_3Br$) crystallizes in the monoclinic space group $P2_1/c$ with $Z=4$. The unit-cell parameters are $a=9.06$, $b=12.66$, $c=16.66$ Å, and $\beta=113.75^\circ$. 2173 reflections were recorded diffractometrically (Cu $K\alpha$ radiation) and used in the determination. The structure was solved by the heavy-atom technique and refined by the method of least squares to a final value of $R=0.059$.

Acronycine, an alkaloid from *Acronychia baueri* Schott which exhibits potent broad spectrum antitumor activity, was initially considered to have either the angular structure (I) or the linear alternative (II) (Drummond & Lahey, 1949). Since chemical degradation of acronycine produced a dimethoxy acid which was not identical with the independently synthesized dimethoxy acid (III) expected to result from degradation of structure (II), Macdonald & Robertson (1966) concluded that acronycine has structure (I). Govindachari, Pai & Subramaniam (1966) also decided in favor of the angular formulation from a consideration of the nuclear magnetic resonance spectrum of a hydrogenated derivative of acronycine.



Our three-dimensional single-crystal X-ray analysis of bromdihydroacronycine (IV), the major product from the reaction of 11,12-dihydroacronycine with *N*-bromosuccinimide,* confirms directly the structural assignment of acronycine as (I).

Slow crystallization of bromdihydroacronycine from ethyl acetate afforded yellow, acicular crystals. The cell parameters and symmetry, obtained from cali-

* We wish to thank Drs James Beck and Albert Pohland of the Eli Lilly Co., Indianapolis, Indiana, for their gift of bromdihydroacronycine. Their recently reported total synthesis of acronycine (Beck, Booher, Brown, Kwok & Pohland, 1967; Beck, Kwok, Booher, Brown, Patterson, Pranc, Rockey & Pohland, 1968) also confirms the structural assignment.

$$c = 16.66 \pm 0.02 \quad Z = 4$$

$$\rho_{calc} = 1.52 \text{ g.cm}^{-3} \quad \rho_{obs} = 1.49 \text{ g.cm}^{-3} \text{ (by flo-}$$

$$\mu = 36.78 \text{ cm}^{-1} \text{ (for Cu } K\alpha \lambda = 1.542 \text{ \AA)} \text{ tation)}$$

Diffraction data were collected from two crystals: crystal I (0.30 × 0.15 × 0.10 mm) was mounted on the *b* axis and crystal II (0.29 × 0.16 × 0.18 mm) was mounted on the elongated *a* axis. Levels *h0l-h8l* were collected from crystal I and levels *0kl-4kl* from crystal II. Intensity measurements were made at 19°C on a Buerger automatic diffractometer measuring backgrounds (B_1 and B_2 counts) for half the time of the main scan (C counts). The angle ω scanned for each reflection was $\omega = [2.0 + (0.8/L)]^\circ$ where $1/L$ is the Lorentz factor. The scan speed was 2° min^{-1} .

Intensities, defined as $I = C - B_1 - B_2$, were not used in the structure analysis if $I \leq 3[C + B_1 + B_2]^{1/2}$, the maximum probable error in the count. All other data were adjusted for Lorentz and polarization factors, and absorption corrections were applied using a local modification of Burnham's (1966) program *GNABS*. Transmission factors ranged from 0.42 to 0.74 for crystal I and from 0.58 to 0.68 for crystal II.

The 14 levels were merged to a common scale (Rae, 1965), and the resulting 2173 independent reflections were placed approximately on an absolute scale using Wilson's (1942) statistical method which indicated an isotropic temperature factor $B = 3.8 \text{ \AA}^2$.

The coordinates of the bromine atom were readily obtained from a sharpened Patterson synthesis and used to compute the first electron density synthesis. Despite the rather high value of the discrepancy index ($R = 0.49$) computed on the basis of bromine contributions only, the twenty-four additional non-hydrogen atoms consistent with structure (IV) were clearly evident. This trial structure ($R = 0.39$) was substantiated completely through further refinement.

The least-squares refinements and subsequent calculations were carried out using the *CRYRM* system of integrated crystallographic routines (Marsh, 1964). The function which was minimized in all cases was $\sum w(|F_o|^2 - |F_c|^2)^2$, where the weighting scheme was as follows:

$$\begin{aligned} \text{if } F_o^2 \geq 300.0 & \quad 1/w = F_o^2/10.0, \\ \text{if } F_o^2 < 300.0 & \quad 1/w = 30.0. \end{aligned}$$

The atomic scattering factor curves employed were those listed in *International Tables for X-ray Crystallography* (1962). The values for bromine were modified for the real part of the dispersion correction ($\Delta f'_{\text{Br}} = -0.95e$).

Four cycles of full-matrix least-squares refinement in which a scale factor, positional parameters, and isotropic temperature factors were varied, resulted in a discrepancy index of $R_F = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.142$ and a weighted index of $R_{wF2} = \sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^4 = 0.135$. After seven more cycles of refinement in which the temperature factor for bromine was re-

financed anisotropically, the R values converged to $R_F = 0.088$ and $R_{wF2} = 0.056$.

A difference-Fourier synthesis at this point indicated anisotropic thermal behavior for many atoms. Peaks ranging from 0.35 to 0.60 e. \AA^{-3} were also found in locations corresponding to the non-methyl hydrogen atom positions. Inclusion of these 8 hydrogen atoms (4 aromatic and 4 methylene hydrogen atoms on the saturated ring), each with $B = 1.5 \text{ \AA}^2$, in a structure factor calculation immediately decreased the discrepancy indices to $R_F = 0.086$ and $R_{wF2} = 0.055$.

Refinement was then continued using anisotropic thermal parameters for all 25 non-hydrogen atoms. Since there were 226 variables, it was necessary to partition them into three matrices which were refined in a single cycle. Matrix I contained the scale factor and positional and anisotropic thermal parameters for the bromine atom. Matrices II and III contained the corresponding parameters for atoms 2-13 and 14-25 respectively. The 8 non-methyl hydrogen atoms were included in their calculated positions ($B = 1.5 \text{ \AA}^2$) but were not allowed to refine. After four cycles of anisotropic refinement, the discrepancy indices converged to their final values of $R_F = 0.059$ and $R_{wF2} = 0.027$. The observed and calculated structure factors are listed in Table 1.

A final difference-Fourier synthesis showed peaks in the vicinity of methyl groups ranging from 0.3 to 0.7 e. \AA^{-3} . However, the proper symmetry for methyl hydrogen atoms could not be established due, most likely, to greater thermal motion of these atoms. There were no other distinctive features on the map.

Discussion of the structure

Fractional coordinates and anisotropic thermal parameters are listed in Tables 2 and 3. Bond distances, angles, and least-squares estimated errors are given in Table 4. Average errors for the bond types C-C, C-O, and C-N, are 0.009, 0.007, 0.008 \AA respectively. Variations within the relatively few justifiable internal comparisons of bond distances and angles in (IV), as well as the average deviation from the least-squares plane II of the aromatic ring, are consistent with the individual least-squares estimated standard deviations.

Table 2. Fractional atomic coordinates

E.s.d.'s ($\times 10^4$) are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>
Br(1)	0.17135 (0.8)	0.22087 (0.5)	0.08709 (0.4)
C(2)	0.2053 (6)	0.0878 (4)	0.0466 (3)
C(3)	0.2289 (6)	0.0017 (4)	0.0984 (3)
O(4)	0.2226 (4)	0.0128 (3)	0.1786 (2)
C(5)	0.2535 (6)	-0.0979 (4)	0.0687 (3)
C(6)	0.2660 (6)	-0.1939 (5)	0.1186 (3)
O(7)	0.2828 (6)	-0.1961 (4)	0.1957 (3)
C(8)	0.2514 (6)	-0.2935 (5)	0.0714 (4)
C(9)	0.2280 (9)	-0.3868 (5)	0.1072 (5)
C(10)	0.2234 (12)	-0.4803 (7)	0.0656 (7)
C(11)	0.3803 (8)	0.0239 (6)	0.2496 (4)

Table 2 (cont.)

	x	y	z
C(12)	0.2442 (12)	-0.4815 (6)	-0.0114 (7)
C(13)	0.2685 (9)	-0.3897 (6)	-0.0489 (5)
C(14)	0.2695 (7)	-0.2936 (5)	-0.0071 (4)
N(15)	0.2928 (6)	-0.1993 (4)	-0.0445 (3)
C(16)	0.2591 (6)	-0.1040 (4)	-0.0149 (3)
C(17)	0.4015 (8)	-0.2030 (6)	-0.0902 (4)
C(18)	0.2286 (6)	-0.0146 (4)	-0.0692 (3)
C(19)	0.2016 (6)	0.0801 (4)	-0.0375 (3)
O(20)	0.1678 (5)	0.1723 (3)	-0.0825 (2)
C(21)	0.1705 (6)	0.1760 (5)	-0.1689 (3)
C(22)	0.0994 (7)	0.0728 (5)	-0.2153 (3)
C(23)	0.1999 (7)	-0.0200 (5)	-0.1651 (3)
C(24)	0.0637 (8)	0.2699 (6)	-0.2117 (5)
C(25)	0.3426 (7)	0.1944 (6)	-0.1576 (4)

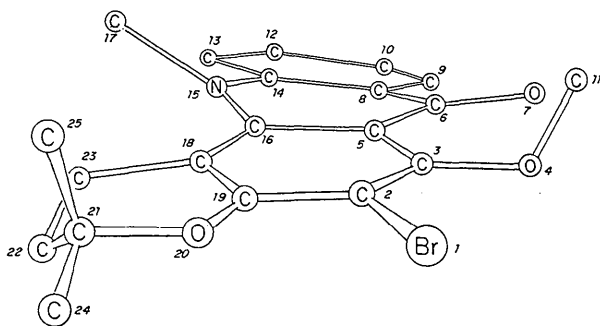


Fig. 1. The molecular conformation of bromdihydroacronycine in the crystal.

Table 4. Bond distances (e.s.d.'s $\times 10^3$ in parentheses) and angles (e.s.d.'s $\times 10$ in parentheses)

Bond	Distance	Bond	Angle
1-2	1.886 (5) Å	1-2-3	119.9 (4) °
2-3	1.354 (7)	1-2-19	118.8 (4)
2-19	1.391 (7)	3-2-19	121.3 (5)
3-4	1.367 (6)	2-3-4	118.8 (5)
3-5	1.406 (8)	2-3-5	120.3 (4)
4-11	1.448 (8)	4-3-5	120.8 (4)
5-6	1.452 (8)	3-4-11	113.1 (4)
5-16	1.416 (7)	3-5-6	122.4 (4)
6-7	1.232 (7)	3-5-16	118.0 (5)
6-8	1.464 (8)	6-5-16	114.5 (5)
8-9	1.377 (9)	5-6-7	124.4 (5)
8-14	1.382 (9)	5-6-8	116.4 (5)
9-10	1.363 (11)	7-6-8	119.2 (5)
10-12	1.370 (15)	6-8-9	120.2 (5)
12-13	1.379 (12)	6-8-14	119.3 (6)
13-14	1.402 (10)	9-8-14	120.4 (6)
14-15	1.402 (8)	8-9-10	120.3 (8)
15-16	1.384 (7)	9-10-12	119.8 (8)
15-17	1.468 (9)	10-12-13	121.5 (8)
16-18	1.406 (7)	12-13-14	118.5 (7)
18-19	1.372 (8)	8-14-13	119.5 (6)
18-23	1.512 (7)	8-14-15	121.1 (5)
19-20	1.355 (7)	13-14-15	119.4 (6)
20-21	1.450 (6)	14-15-16	119.4 (5)
21-22	1.523 (9)	14-15-17	117.2 (5)
21-24	1.518 (10)	16-15-17	120.2 (5)
21-25	1.512 (9)	5-16-15	119.9 (5)
22-23	1.516 (9)	5-16-18	120.9 (5)
		15-16-18	119.2 (4)
		16-18-19	118.3 (4)
		16-18-23	123.3 (5)
		19-18-23	117.8 (5)
		2-19-18	121.0 (5)

Table 3. Anisotropic temperature factors and their estimated standard deviations ($\times 10^4$)

Thermal parameters are of the form

$$T = \exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)] .$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
1	234 (1)	68.6 (0.5)	44.4 (0.3)	37 (1)	111 (1)	-2.5 (0.6)
2	112 (7)	68 (4)	30 (2)	11 (9)	60 (6)	5 (4)
3	105 (7)	73 (4)	27 (2)	6 (9)	45 (6)	-1 (4)
4	170 (6)	83 (3)	31 (1)	21 (7)	88 (5)	6 (3)
5	100 (7)	68 (4)	33 (2)	-17 (8)	51 (6)	3 (4)
6	98 (7)	72 (4)	42 (2)	7 (9)	36 (7)	11 (5)
7	273 (9)	84 (3)	42 (2)	-16 (9)	105 (7)	28 (4)
8	117 (8)	69 (4)	50 (3)	-1 (7)	38 (7)	7 (5)
9	216 (12)	62 (4)	75 (4)	7 (13)	68 (11)	28 (7)
10	357 (20)	73 (5)	88 (5)	-1 (17)	112 (17)	4 (9)
11	212 (11)	104 (5)	33 (2)	-26 (13)	46 (9)	-20 (6)
12	341 (19)	65 (5)	100 (6)	12 (17)	64 (17)	-45 (9)
13	237 (14)	77 (5)	71 (4)	-3 (14)	70 (12)	-50 (7)
14	147 (9)	70 (4)	50 (3)	-5 (10)	48 (8)	-15 (5)
15	152 (7)	68 (3)	41 (2)	3 (8)	68 (6)	-19 (4)
16	110 (7)	71 (4)	33 (2)	-19 (9)	52 (6)	-8 (5)
17	181 (10)	118 (6)	46 (3)	22 (13)	89 (9)	-35 (6)
18	120 (7)	70 (4)	28 (2)	-25 (9)	51 (6)	-11 (4)
19	130 (8)	74 (4)	29 (2)	-8 (9)	41 (7)	18 (5)
20	185 (7)	72 (3)	33 (2)	8 (7)	68 (5)	17 (3)
21	144 (8)	95 (4)	31 (2)	5 (10)	72 (7)	34 (5)
22	161 (9)	103 (5)	29 (2)	-41 (11)	39 (7)	6 (5)
23	182 (10)	95 (5)	29 (2)	-41 (12)	61 (8)	-29 (5)
24	188 (10)	118 (6)	58 (3)	121 (13)	105 (10)	84 (7)
25	132 (9)	102 (5)	59 (3)	-25 (11)	71 (9)	40 (6)

Table 4 (cont.)

Bond	Angle
2-19-20	113.9 (5)
18-19-20	125.1 (4)
19-20-21	119.1 (4)
20-21-22	107.8 (5)
20-21-24	103.3 (5)
20-21-25	108.1 (4)
22-21-24	112.0 (5)
22-21-25	113.5 (5)
24-21-25	111.5 (6)
21-22-23	110.3 (5)
18-23-22	111.0 (5)

Table 5. Selected dihedral angles*

Atom group	Angle
2-3-4-11	97°
5-3-4-11	85
7-6-5-3	12
7-6-8-14	165
7-6-8-9	11
7-6-5-16	170
17-15-16-5	138
17-15-16-18	43
17-15-14-8	144
17-15-14-13	35
20-19-18-23	7
21-20-19-18	5
22-23-18-19	17
22-21-20-19	38

Fig. 1 is an informative view of the molecular conformation in the crystal matrix while Fig.2 shows the relative magnitudes and orientations of the thermal ellipsoids (Johnson, 1965). The C(11) methyl group is rotated out of the plane of the adjoining ring, thus minimizing overlap with atoms Br(1) and O(7). The dihedral angle between the planes defined by atoms C(2)-C(3)-O(4) and atoms C(3)-O(4)-C(11) is 97° (Table 5). Likewise, the angle between planes defined by atoms C(5)-C(3)-O(4) and C(3)-O(4)-C(11) is 85°.

The nitrogen heterocyclic ring is slightly bent in a boat conformation. The dihedral angle between the plane defined by atoms C(14), N(15), and C(16), plane

* The dihedral angle is the angle between the planes defined by atoms *a, b, c*, and atoms *b, c, d*, of atom group *a, b, c, d*.

IV (Table 6), and the least-squares plane of atoms C(5), C(8), C(14), and C(16) (plane V) is 16°. Similarly, the dihedral angle between plane III [atoms C(5), C(6), O(7), and C(8)] and plane V is 11°, while the angle between planes III and IV is 27°. The methyl carbon atom C(17) is somewhat displaced from the plane of atoms C(14), N(15), and C(16) and away from plane V. The angle between plane IV and the N(15)-C(17) bond is 18°.

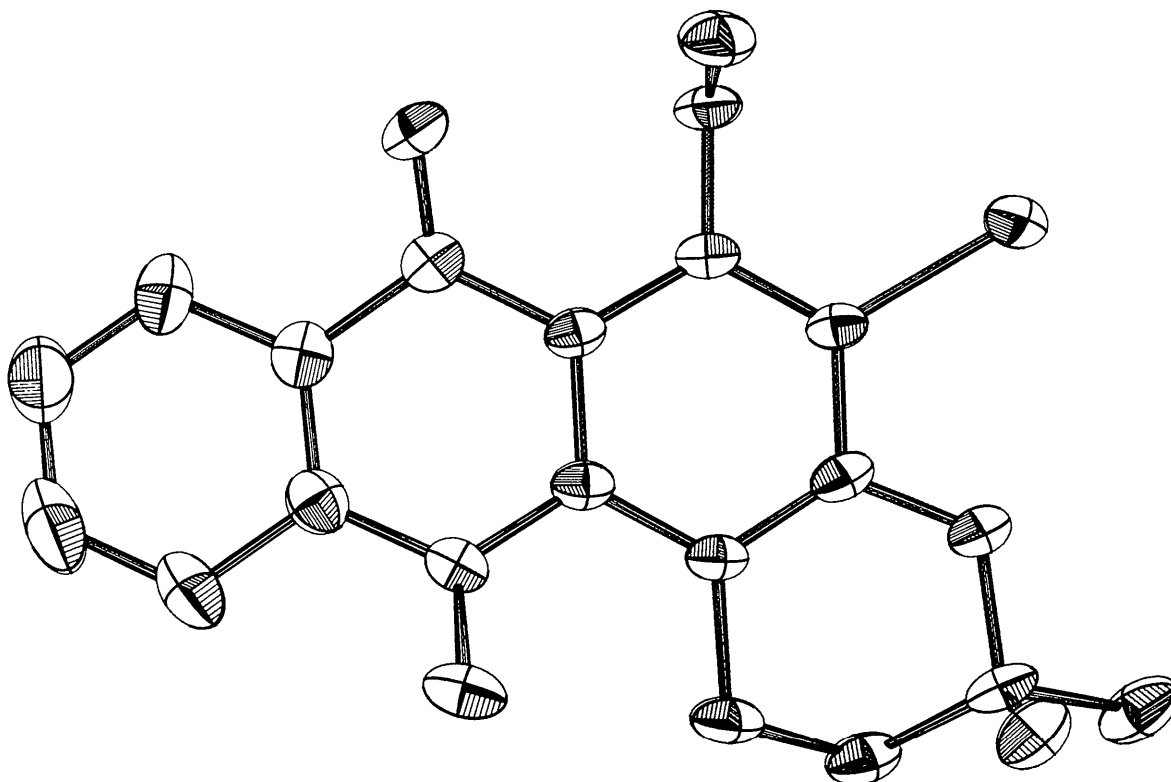


Fig.2. The 30% probability contour of the thermal ellipsoids.

Table 6. *Important planes in bromdihydroacronycine*Plane I: $0.9044X + 0.1830Y + 0.3853Z = 1.8593$ †

Deviation		Deviation	
Br(1)	0.04 Å	N(15)	0.09 Å
C(2)†	0.02	C(16)†	0.03
C(3)†	0.00	C(17)	0.98
O(4)	-0.04	C(18)†	-0.01
C(5)†	-0.02	C(19)†	-0.01
C(6)	-0.15	O(20)	-0.07
O(7)	-0.03	C(23)	0.24

Plane II: $0.8562X - 0.0912Y + 0.5084Z = 2.4271$ †

Deviation		Deviation	
C(6)	0.10 Å	C(12)†	0.00 Å
O(7)	0.39	C(13)†	0.01
C(8)†	0.01	C(14)†	-0.01
C(9)†	0.00	N(15)	-0.01
C(10)†	-0.01	C(17)	0.74

Plane III: $0.9588X - 0.0100Y + 0.2837Z = 2.0738$ †

Deviation		Deviation	
C(5)†	0.00 Å	O(7)†	0.00 Å
C(6)†	0.01	C(8)†	0.00

Plane IV: $0.7256X + 0.0468Y + 0.6864Z = 1.5581$ †

Deviation		Deviation	
C(14)†	—	C(16)†	—
N(15)†	—	C(17)	0.46 Å

Plane V: $0.8850X + 0.0157Y + 0.4652Z = 2.0664$ †

Deviation		Deviation	
C(2)	-0.35 Å	C(12)	-0.22 Å
C(3)	-0.12	C(13)	-0.05
C(5)†	0.03	C(14)†	0.03
C(6)	0.17	N(15)	0.19
O(7)	0.39	C(16)†	-0.03
C(8)†	-0.03	C(17)	1.01
C(9)	-0.19	C(18)	-0.32
C(10)	-0.29	C(19)	-0.48

† Planes are defined in terms of the orthonormal axes X , Y , Z , which are directed along the crystallographic axes a , b , and c^* respectively.

These atoms were used to calculate the plane.

Each of the aromatic rings is planar within experimental error (planes I and II); they are not, however, parallel. Corresponding to the displacement of N(15) and C(6) above plane V [atoms C(5), C(8), C(14), C(16)], there is a slight downward tilt of both planes I and II. They form dihedral angles of 11 and 7° respectively, with plane V.

The bromine atom is displaced slightly (+0.04 Å) from the plane I of the aromatic ring so as to minimize overlap with the two *ortho* substituents [O(4) and O(20)] which are displaced correspondingly to the opposite side of the aromatic ring by -0.04 and -0.07 Å, respectively. A significant reduction of the cyclic C-N bond lengths due to zwitterionic vinylous amide resonance forms, as observed in α -pyridone (Penfold, 1953), is not apparent in (IV).

Molecular packing

With the exception of the methyl groups and C(22), the molecules are approximately flat and are arranged in close-packed layers parallel to (100) in the crystal lattice. Each close-packed layer (Fig. 3) contains chains parallel to c consisting of alternating enantiomeric molecules, related by the c -glide operation. Simple chain translation parallel to [010] completes the layer and defines the lattice parameter b .

The displacement of the methyl groups and C(22) from the 'flat' portion of the molecule results in the nonequivalence of opposite sides of a given layer. When the layers are stacked with inversion symmetry, an operation which completes the space group symmetry, two different interlayer distances (4.6 and 3.7 Å) result.*

Interatomic distances across the 4.6 Å layer spacings between the 'protruding' methyl carbon atoms and other atoms of the adjacent layer are shown in Fig. 4. The intermolecular contacts within the close-packed layers are of the order of the sum of the van der Waals radii.

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* In order to determine these 'interlayer spacings', the flat portion of the molecule was approximated by a least-squares plane through the twelve benzenoid carbon atoms. The distances between these 'molecular planes' in successive layers are 3.7 and 4.6 Å.

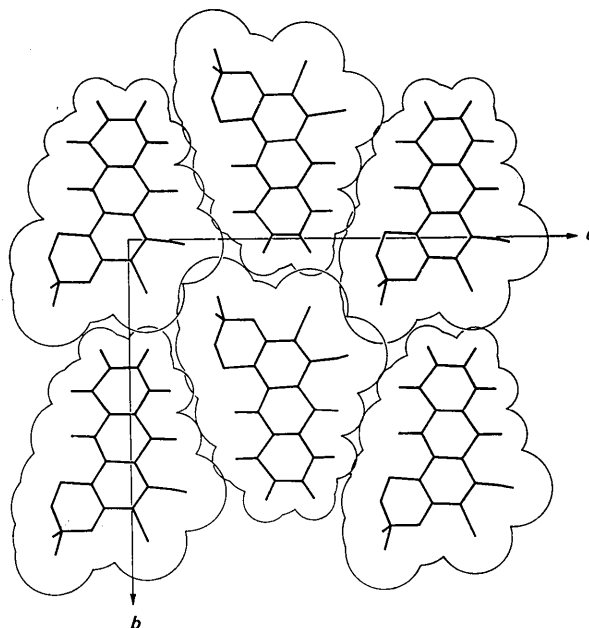


Fig. 3. Close-packed layer parallel to (100).

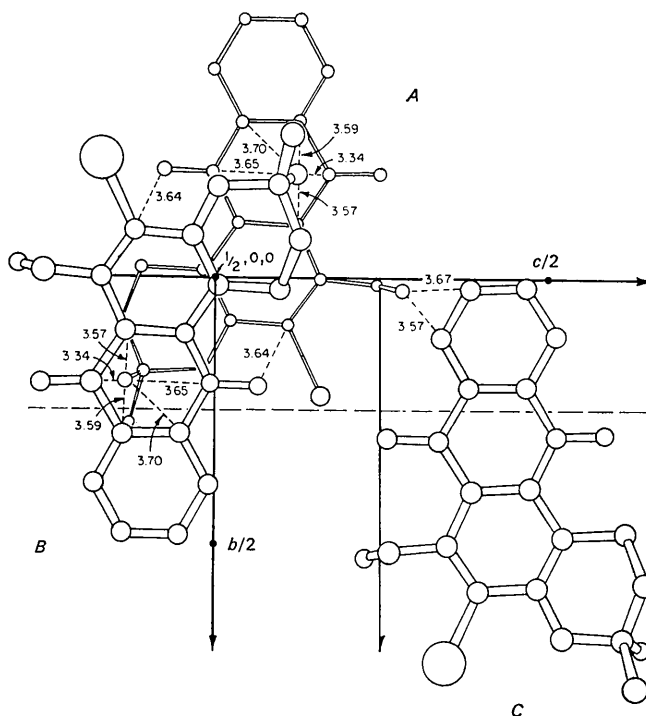


Fig.4. (100) projection showing contacts between layers spaced 4.6 Å apart. Molecules pictured are (A), x, y, z ; (B) $\bar{x}+1, \bar{y}, \bar{z}$; (C) $\bar{x}+1, \frac{1}{2}+y, \frac{1}{2}-z$

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