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## The Crystal and Molecular Structure of 1-(Diphenylmethyl)azetidin-3-ol

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1-(Diphenylmethyl)azetidin-3-ol is triclinic, space group  $P\bar{1}$ , with  $a = 8.479(2)$ ,  $b = 17.294(4)$ ,  $c = 10.606(3)$  Å,  $\alpha = 118.59(2)^\circ$ ,  $\beta = 100.30(2)^\circ$ ,  $\gamma = 89.63(2)^\circ$ ,  $Z = 4$ . The structure was solved by multi-solution methods and refined to an  $R$  of 0.044 for 2755 reflexions. The four-membered rings in the two independent molecules are puckered with dihedral angles of 156 and 153°. The two molecules differ in conformation with respect to rotation of the phenyl rings about the C–C bonds. The structure is stabilized by a network of O–H...N intermolecular hydrogen bonds.

### Introduction

A fundamental assumption in the analysis of structure–activity relationships is that the structure of the substrate molecule is complementary to that of the receptor. In order to obtain information about the mode of binding of acetylcholine and related compounds at its cholinergic receptor, X-ray investigations of many cholinergic agents have been carried out (Baker, Chothia, Pauling & Petcher, 1971). During the synthesis of rigid or semi-rigid analogues of acetylcholine, Chatterjee & Triggler (1968) obtained the title compound and an X-ray investigation was undertaken to establish the conformation and stereochemistry. The problem of the geometry of four-membered rings is a fascinating one. For example, the cyclobutane ring is found to occur in both planar and puckered forms (Moriarty, 1974; Cotton & Frenz, 1974). Results on the azetidine ring are limited and the title compound provides an opportunity to study its geometry.

### Experimental

Crystals were supplied by Dr S. Chatterjee. That used for data collection was  $0.3 \times 0.2 \times 0.6$  mm. The data were collected at Wisconsin on a Picker FACS-I

computer-controlled diffractometer with Cu  $K\alpha$  radiation. The intensities were measured in the  $\theta$ – $2\theta$  scan mode at a scan rate of  $2^\circ \text{ min}^{-1}$  and a scan width of  $(2 + 0.285 \tan \theta)^\circ$  for  $4^\circ \leq 2\theta \leq 125^\circ$ . The agreement between 600 inverse reflexions [ $2\sum |F_1 - F_2| / \sum (F_1 + F_2)$ ] was 0.02. The cell constants were determined by a least-squares fit on 12 reflexions with  $40^\circ \leq 2\theta \leq 50^\circ$ . Crystal data are summarized in Table 1. The final data set contained 3845 independent reflexions excluding the reflexions for which the net count was less than the net background. Reflexions with  $F_o < 2.0\sigma(F_o)$  were treated as unobserved. The data were corrected for the Lorentz–polarization factor but not for absorption ( $\mu r < 0.30$ ).

Table 1. *Crystal data*

$C_{16}H_{17}NO$	F.W. 239.32
Triclinic	$Z = 4$
Space group $P\bar{1}$	$F(000) = 512$
$a = 8.479(2)$ Å	$\alpha = 118.59(2)^\circ$
$b = 17.294(4)$	$\beta = 100.30(2)^\circ$
$c = 10.606(3)$	$\gamma = 89.63(2)^\circ$
$V = 1338(2)$ Å <sup>3</sup>	$\lambda(\text{Cu } K\alpha) = 1.5418$ Å
$D_m = 1.24$ g cm <sup>-3</sup>	$D_x = 1.188$ g cm <sup>-3</sup>
$\mu(\text{Cu } K\alpha) = 4.93$ cm <sup>-1</sup>	

## Structure determination and refinement

The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) adapted to an IBM 360/44

computer with 64 Kbyte memory by Ramakumar & Narasimha Murthy (1974). *E* values were calculated with the molecular scattering factors for the four-membered and phenyl rings. Five symbols were chosen and

Table 2. Final parameters and their *e.s.d.*'s (in parentheses)

For the heavy atoms, the coordinates have been multiplied by  $10^5$  and the  $\beta_{ij}$  by  $10^4$ ; the coordinates of the H atoms are  $\times 10^4$ . The temperature factor is of the form  $\exp[-(\beta_{11}h^2 + \dots + 2hk\beta_{12} + \dots)]$  for the heavy atoms and  $\exp(-B \sin^2\theta/\lambda^2)$  for the H atoms.

	<i>x</i>	<i>y</i>	<i>z</i>	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
O(1A)	31838 (46)	52206 (22)	30996 (41)	217 (7)	44 (2)	178 (6)	29 (3)	-5 (5)	28 (3)
N(1A)	19505 (44)	71242 (23)	34910 (40)	143 (6)	36 (2)	128 (5)	8 (3)	34 (5)	33 (3)
C(1A)	18409 (57)	65670 (30)	42099 (51)	153 (8)	42 (2)	128 (7)	7 (3)	33 (6)	36 (3)
C(2A)	20890 (58)	57528 (29)	28082 (53)	161 (8)	37 (2)	138 (7)	8 (3)	20 (6)	33 (3)
C(3A)	27053 (64)	63999 (32)	23643 (55)	203 (10)	46 (3)	132 (7)	14 (4)	62 (7)	28 (4)
C(4A)	29934 (53)	79542 (28)	43995 (51)	130 (7)	38 (2)	137 (7)	6 (3)	12 (5)	35 (3)
C(5A)	22356 (56)	85996 (27)	56540 (49)	168 (8)	33 (2)	117 (6)	-3 (3)	21 (6)	35 (3)
C(6A)	8204 (62)	89633 (33)	54212 (55)	178 (9)	50 (3)	142 (7)	8 (4)	39 (7)	42 (4)
C(7A)	1846 (69)	95814 (34)	65791 (64)	212 (11)	46 (3)	174 (9)	24 (4)	64 (8)	34 (4)
C(8A)	9480 (83)	98308 (37)	79973 (68)	267 (13)	52 (3)	171 (9)	0 (5)	79 (9)	37 (4)
C(9A)	23258 (87)	84652 (39)	82567 (60)	326 (15)	58 (3)	118 (8)	-11 (6)	30 (8)	33 (4)
C(10A)	29618 (69)	88507 (34)	70883 (57)	224 (10)	51 (3)	144 (8)	3 (4)	15 (7)	51 (4)
C(11A)	34001 (60)	83512 (29)	34931 (54)	174 (9)	36 (2)	146 (7)	11 (3)	49 (6)	35 (3)
C(12A)	22931 (70)	93542 (36)	23601 (61)	219 (10)	61 (3)	171 (8)	22 (4)	65 (8)	60 (4)
C(13A)	26962 (83)	87729 (40)	16169 (65)	290 (14)	67 (3)	186 (9)	45 (6)	95 (9)	70 (5)
C(14A)	41765 (100)	91882 (41)	19432 (76)	347 (17)	61 (3)	198 (11)	11 (6)	120 (11)	57 (5)
C(15A)	52995 (89)	91852 (40)	30442 (81)	287 (14)	58 (3)	234 (12)	-32 (5)	107 (11)	42 (5)
C(16A)	49229 (68)	87694 (36)	38074 (63)	198 (10)	55 (3)	174 (9)	-16 (4)	39 (8)	37 (4)
O(1B)	14054 (42)	29686 (27)	-26762 (35)	193 (6)	99 (3)	110 (5)	18 (3)	12 (4)	51 (3)
N(1B)	34521 (40)	35713 (22)	6760 (37)	121 (5)	44 (2)	111 (5)	1 (2)	8 (4)	34 (2)
C(1B)	18145 (50)	31188 (30)	-1658 (48)	119 (7)	60 (3)	119 (6)	3 (3)	7 (5)	43 (3)
C(2B)	18840 (56)	35233 (32)	-11695 (50)	159 (8)	64 (3)	118 (6)	12 (4)	9 (5)	48 (3)
C(3B)	37042 (56)	36607 (34)	-6074 (51)	157 (8)	65 (3)	150 (7)	-7 (4)	19 (6)	57 (4)
C(4B)	45452 (48)	30265 (24)	10859 (43)	127 (6)	34 (2)	102 (5)	8 (3)	16 (5)	22 (3)
C(5B)	38552 (48)	27876 (26)	20859 (45)	118 (6)	39 (2)	122 (6)	6 (3)	7 (5)	33 (3)
C(6B)	39012 (54)	33923 (29)	35459 (50)	157 (8)	46 (2)	126 (6)	14 (3)	21 (5)	35 (3)
C(7B)	32446 (61)	31554 (37)	44244 (53)	177 (9)	73 (3)	130 (7)	27 (4)	38 (6)	50 (4)
C(8B)	25323 (61)	23142 (40)	38767 (66)	172 (9)	84 (4)	214 (9)	17 (5)	47 (7)	93 (5)
C(9B)	24890 (66)	17065 (35)	24295 (67)	204 (10)	58 (3)	226 (10)	-12 (4)	35 (8)	69 (5)
C(10B)	31425 (59)	19382 (30)	15450 (53)	172 (8)	47 (2)	153 (7)	-4 (4)	22 (6)	37 (4)
C(11B)	62447 (48)	34752 (27)	17488 (43)	118 (6)	44 (2)	102 (5)	10 (3)	21 (5)	28 (3)
C(12B)	66605 (56)	43536 (32)	22391 (62)	128 (8)	44 (2)	227 (9)	3 (3)	16 (7)	29 (4)
C(13B)	82506 (64)	47098 (37)	28559 (69)	140 (9)	58 (3)	246 (10)	-14 (4)	22 (7)	19 (5)
C(14B)	94292 (56)	41932 (42)	29858 (58)	113 (8)	87 (4)	159 (8)	-5 (4)	19 (6)	44 (4)
C(15B)	90334 (56)	33164 (40)	24876 (58)	136 (8)	91 (4)	178 (8)	33 (4)	43 (6)	81 (5)
C(16B)	74595 (54)	29627 (32)	18753 (51)	147 (8)	60 (3)	144 (7)	10 (4)	32 (6)	51 (4)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )		<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
H(1A)	2735 (32)	6734 (17)	5051 (28)	4.5 (0.6)	H(1B)	973 (35)	3298 (18)	461 (31)	5.5 (0.7)
H(2A)	826 (29)	6578 (15)	4551 (26)	3.6 (0.5)	H(2B)	1862 (30)	2446 (16)	-705 (27)	4.2 (0.6)
H(3A)	1023 (32)	5391 (17)	2153 (29)	4.8 (0.6)	H(3B)	1357 (36)	4133 (19)	-840 (32)	5.6 (0.7)
H(4A)	2294 (34)	6296 (18)	1332 (30)	5.1 (0.6)	H(4B)	4259 (39)	3133 (21)	-1303 (35)	6.8 (0.8)
H(5A)	3977 (38)	6484 (20)	2602 (35)	6.4 (0.8)	H(5B)	4330 (34)	4214 (18)	-403 (30)	5.3 (0.7)
H(6A)	4027 (31)	7833 (17)	4816 (28)	4.2 (0.6)	H(6B)	4606 (30)	2475 (16)	182 (26)	3.9 (0.6)
H(7A)	299 (34)	8824 (18)	4427 (30)	5.1 (0.6)	H(7B)	4438 (32)	3985 (17)	3929 (29)	4.4 (0.6)
H(8A)	-864 (41)	9833 (21)	6316 (36)	7.2 (0.8)	H(8B)	3308 (36)	3581 (19)	5418 (33)	5.9 (0.7)
H(9A)	488 (42)	10279 (22)	8807 (38)	7.5 (0.9)	H(9B)	2064 (44)	2139 (23)	4494 (39)	8.3 (1.0)
H(10A)	2882 (43)	9644 (22)	9294 (38)	7.7 (0.9)	H(10B)	1988 (42)	1082 (22)	1989 (38)	7.4 (0.9)
H(11A)	3958 (36)	8599 (19)	7193 (32)	5.7 (0.7)	H(11B)	3066 (35)	1523 (18)	500 (31)	5.3 (0.7)
H(12A)	1261 (36)	8036 (19)	2139 (32)	5.7 (0.7)	H(12B)	5832 (37)	4758 (20)	2154 (33)	6.3 (0.8)
H(13A)	1885 (41)	8739 (22)	841 (36)	7.4 (0.8)	H(13B)	8488 (47)	5324 (25)	3234 (41)	8.9 (1.0)
H(14A)	4544 (48)	9520 (25)	1398 (42)	9.4 (1.0)	H(14B)	10595 (39)	4456 (21)	3400 (35)	6.6 (0.8)
H(15A)	6336 (45)	9436 (23)	3312 (39)	8.3 (0.9)	H(15B)	9861 (42)	2948 (22)	2559 (38)	7.8 (0.9)
H(16A)	5663 (36)	8764 (19)	4569 (32)	5.6 (0.7)	H(16B)	7144 (31)	2352 (17)	1544 (28)	4.3 (0.6)
H(17A)	3189 (47)	4707 (24)	2221 (42)	8.9 (1.0)	H(17B)	158 (58)	2916 (29)	-2929 (49)	11.7 (1.3)

the non-trivial set whose combined figure-of-merit was the highest produced an  $E$  map from which all 36 non-hydrogen atoms corresponding to the two independent molecules could be located. The parameters were initially refined with a block-diagonal LS program of Shiono (1968) adapted by Dr B. S. Reddy. The H atoms were located in a difference map when  $R$  was 11.5%. The final refinement was carried out with the program written by Busing, Martin & Levy (1962) adapted by S. T. Rao for the Univac 1110 computer. The quantity minimized was  $\sum w(|F_o| - |F_c|)^2$  where the weight  $w$  is defined by  $w = 1.0/(2.0 + 0.0271|F_o|)^2$  for  $|F_o| < 120.0$  and  $w = 1.0/(5.25)^2$  for  $|F_o| > 120.0$ .  $R$  for reflexions with  $|F_o| < 2.80$  was 70%, which necessitated the exclusion of weak reflexions from the refinement. Fourteen reflexions which were affected by secondary extinction were also excluded. Full-matrix refinement was performed with the parameters of one molecule in one pass and those of the other in a second pass. The coordinates and anisotropic temperature factors were refined for the non-hydrogen atoms and coordinates and isotropic temperature factors for H. The final  $R$  was 0.044 for 2755 reflexions with the standard deviation of an observation of unit weight of 0.89.  $R$  for the entire set of 3845 reflexions was 0.064. The final positional and thermal parameters are listed in Table 2. Scattering factors for non-hydrogen atoms were those from *International Tables for X-ray Crystallography* (1962). For H the values were from Stewart, Davidson & Simpson (1965).\*

### Discussion

The packing of the molecules in the crystal is depicted in Fig. 1. The molecules are packed so that there is a

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32114 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

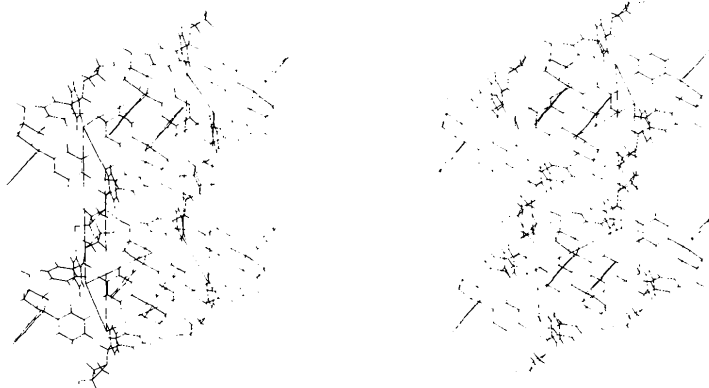


Fig. 1. Stereo diagram of the molecular packing.

clustering of six phenyl rings at 0,0,0 and four at  $0,0,\frac{1}{2}$ . The four-membered rings, which contain the active polar groups, cluster around the centre at  $0,\frac{1}{2},0$  resulting in a network of hydrogen bonds. The hydrogen bonds are of the type  $O-H \cdots N$ , the details of which are given in Table 3. The proton associated with the OH group of molecule  $A$  is accepted by the lone pair of the N atom of molecule  $B$  and *vice versa*. As expected the shorter hydrogen bond is found to be more linear. The only other intermolecular contact which is somewhat short is  $O(1B) \cdots C(1A)$  (3.239 Å). This arises because  $O(1B)$  hydrogen-bonds with  $N(1A)$  by approaching the ring in a direction such that it forms a short contact with  $C(1A)$  of the four-membered ring. However, the packing is not intimate, as indicated by the low density of  $1.188 \text{ g cm}^{-3}$ .

The  $N \cdots O$  intramolecular distances in molecules  $A$  and  $B$  are 3.303 and 3.318 Å respectively and this feature is dictated by the ring geometry. The  $N^+ \cdots O$  distance is of the order of 3.29 Å in acetylcholine in both the solid and gas phases (Baker *et al.*, 1971). The conformational angles, as defined by Baker *et al.* (1971), for the title compound are  $\tau_1 = 138.1$  and  $\tau_2 = 135.9^\circ$ . The angles for potent muscarinic activity are  $\tau_1 = 180^\circ$  and  $\tau_2$  from  $73$  to  $137^\circ$ , suggesting that the compound investigated by us may be expected to be muscarinically inactive.

Bond distances and angles involving C, N and O for the two molecules are shown in Table 4. The thermal ellipsoids for molecules  $A$  and  $B$  are depicted in Fig. 2. The chemically equivalent bond lengths and angles in

Table 3. Details of the hydrogen bonds,  $D-H \cdots A$

$D$	$H$	$A$	$D \cdots A$	$H \cdots A$	$\angle D-H \cdots A$
$O(1A)-H(17A) \cdots N(1B)^i$			2.825 Å	1.91 Å	$168^\circ$
$O(1B)-H(17B) \cdots N(1A)^j$			2.804	1.77	174

Symmetry code: (i)  $x, y, z$ ; (ii)  $-x, 1-y, -z$ .

Table 4. Bond distances and angles involving the non-hydrogen atoms

The estimated standard deviations are in parentheses.

	Molecule A	Molecule B		Molecule A	Molecule B
C(1)—C(2)	1.530 (6) Å	1.537 (10) Å	C(8)—C(9)	1.372 (10) Å	1.378 (8) Å
C(1)—N(1)	1.499 (8)	1.497 (5)	C(9)—C(10)	1.388 (8)	1.377 (11)
C(2)—O(1)	1.401 (7)	1.395 (5)	C(5)—C(10)	1.385 (8)	1.390 (7)
C(2)—C(3)	1.532 (10)	1.528 (7)	C(4)—C(11)	1.506 (10)	1.518 (6)
C(3)—N(1)	1.496 (6)	1.493 (8)	C(11)—C(12)	1.388 (9)	1.374 (7)
N(1)—C(4)	1.469 (5)	1.474 (7)	C(12)—C(13)	1.381 (12)	1.391 (7)
C(4)—C(5)	1.518 (7)	1.511 (8)	C(13)—C(14)	1.353 (11)	1.369 (9)
C(5)—C(6)	1.389 (8)	1.386 (6)	C(14)—C(15)	1.371 (12)	1.367 (10)
C(6)—C(7)	1.384 (8)	1.378 (10)	C(15)—C(16)	1.388 (13)	1.377 (7)
C(7)—C(8)	1.379 (9)	1.377 (9)	C(11)—C(16)	1.389 (8)	1.385 (7)
O(1)—C(2)—C(1)	112.2 (4)°	117.5 (4)°	C(4)—C(11)—C(16)	120.2 (4)°	117.6 (4)°
O(1)—C(2)—C(3)	118.1 (4)	114.3 (4)	C(5)—C(6)—C(7)	121.4 (5)	120.6 (4)
N(1)—C(1)—C(2)	89.5 (4)	89.0 (4)	C(6)—C(5)—C(10)	117.7 (5)	118.0 (4)
C(1)—N(1)—C(3)	89.2 (4)	88.8 (4)	C(5)—C(10)—C(9)	121.2 (5)	121.0 (5)
N(1)—C(3)—C(2)	89.5 (4)	89.5 (4)	C(6)—C(7)—C(8)	119.6 (5)	120.9 (5)
C(1)—N(1)—C(4)	114.7 (4)	114.6 (4)	C(7)—C(8)—C(9)	120.1 (6)	118.9 (5)
C(3)—N(1)—C(4)	114.4 (4)	116.4 (4)	C(8)—C(9)—C(10)	119.9 (6)	120.5 (5)
N(1)—C(4)—C(5)	110.7 (4)	109.5 (4)	C(11)—C(12)—C(13)	120.9 (5)	120.5 (5)
N(1)—C(4)—C(11)	111.4 (4)	111.9 (4)	C(12)—C(11)—C(16)	116.6 (5)	117.8 (4)
C(1)—C(2)—C(3)	86.7 (4)	86.1 (4)	C(11)—C(16)—C(15)	121.4 (5)	121.6 (5)
C(4)—C(5)—C(6)	122.1 (4)	122.1 (4)	C(12)—C(13)—C(14)	122.0 (6)	120.7 (5)
C(4)—C(5)—C(10)	120.1 (4)	119.9 (4)	C(13)—C(14)—C(15)	118.3 (6)	119.3 (5)
C(5)—C(4)—C(11)	111.9 (4)	112.6 (4)	C(14)—C(15)—C(16)	120.8 (6)	120.0 (5)
C(4)—C(11)—C(12)	123.1 (4)	124.7 (4)			

Table 5. Some planes of interest

An asterisk indicates atoms included in the calculation of the least-squares plane.

## Molecule A

Equations of planes (Cartesian coordinates)

(a)  $0.8510X - 0.0655Y + 0.5211Z = 2.000$

(b)  $0.7235X - 0.1260Y + 0.6787Z = 1.607$

(c)  $0.8082X + 0.1394Y + 0.5722Z = 4.159$

(d)  $0.9397X + 0.0023Y + 0.3420Z = 2.120$

(e)  $0.8552X - 0.2547Y + 0.4514Z = 0.099$

Deviations of atoms (Å)

	(a)	(b)	(c)	(d)	(e)
C(1A)	0.113*	0.442	0.000*	0.000*	0.000*
C(2A)	-0.110*	0.000*	-0.452	0.000*	0.000*
C(3A)	0.113*	0.000*	0.000*	0.442	0.000*
N(1A)	-0.116*	0.000*	0.000*	0.000*	-0.433
C(4A)	0.866	0.962	1.193	0.963	0.310
O(1A)	0.836	0.942	0.252	0.903	1.133

## Molecule B

Equations of planes

(a)  $-0.3614X + 0.7281Y + 0.5825Z = 3.448$

(b)  $-0.1568X + 0.8416Y + 0.5168Z = 4.783$

(c)  $-0.4635X + 0.8045Y + 0.3713Z = 3.607$

(d)  $-0.5453X + 0.5762Y + 0.6088Z = 2.189$

(e)  $-0.2461X + 0.6180Y + 0.7467Z = 2.877$

Deviations of atoms (Å)

	(a)	(b)	(c)	(d)	(e)
C(1B)	-0.128*	-0.503	0.000*	0.000*	0.000*
C(2B)	0.126*	0.000*	0.514	0.000*	0.000*
C(3B)	-0.130*	0.000*	0.000*	-0.499	0.000*
N(1B)	0.132*	0.000*	0.000*	0.000*	0.490
C(4B)	-0.792	-0.907	-1.177	-0.895	-0.149
O(1B)	-0.772	-0.856	-0.096	-0.881	-1.117

the independent molecules are not significantly different. The angles within the four-membered ring are close to  $90^\circ$  with  $C(1)-C(2)-C(3)$  smaller [ $86.4(4)^\circ$ ] than the others. From the calculations of the planes

through the various combinations of three atoms of the four-membered ring,  $C(2)$  shows the largest deviation from the plane through  $N(1)$ ,  $C(1)$  and  $C(3)$  in molecules *A* and *B* (Table 5). The endocyclic  $N-C$  bonds [ $1.496(7) \text{ \AA}$ ] are longer than the exocyclic ones [ $1.472(6) \text{ \AA}$ ]. This lengthening may be attributed to the strain in the four-membered ring. The angles  $C(5A)-C(4A)-C(11A)$  [ $111.9(4)^\circ$ ] and  $C(5B)-C(4B)-C(11B)$  [ $112.6(4)^\circ$ ] are slightly greater than the tetrahedral value owing to the steric interaction between the bulky groups attached to  $C(4)$ . The angles  $C(4A)-C(5A)-C(6A)$  [ $122.1(4)^\circ$ ],  $C(4A)-C(11A)-C(12A)$  [ $123.1(4)^\circ$ ],  $C(4B)-C(5B)-C(6B)$  [ $122.1(4)^\circ$ ] and  $C(4B)-C(11B)-C(12B)$  [ $124.7(4)^\circ$ ] are significantly larger than  $C(4A)-C(5A)-C(10A)$  [ $120.1(4)^\circ$ ],  $C(4A)-C(11A)-C(16A)$  [ $120.2(4)^\circ$ ],  $C(4B)-C(5B)-C(10B)$  [ $119.9(4)^\circ$ ] and  $C(4B)-C(11B)-C(16B)$  [ $117.6(4)^\circ$ ] respectively, because  $C(6A)$ ,  $C(12A)$ ,  $C(6B)$  and  $C(12B)$  are nearer to atoms of the four-membered ring than  $C(10A)$ ,  $C(16A)$ ,  $C(10B)$  and  $C(16B)$ .  $C(2A)-O(1A)$  [ $1.401(7) \text{ \AA}$ ] and  $C(2B)-O(1B)$  [ $1.395(5) \text{ \AA}$ ] are comparable with the  $C-O$  length of  $1.409(9) \text{ \AA}$  observed in *N*-methyl-*N*-*tert*-butyl-3-hydroxyazetidinium methanesulphonate (McGandy, Berman, Burgner & Van Etten, 1969). The average value of the endocycle  $C-C$  distances [ $1.532(9) \text{ \AA}$ ] is in agreement with the value obtained in *L*-azetidine-2-carboxylic acid (Berman, McGandy, Burgner & Van Etten, 1969).

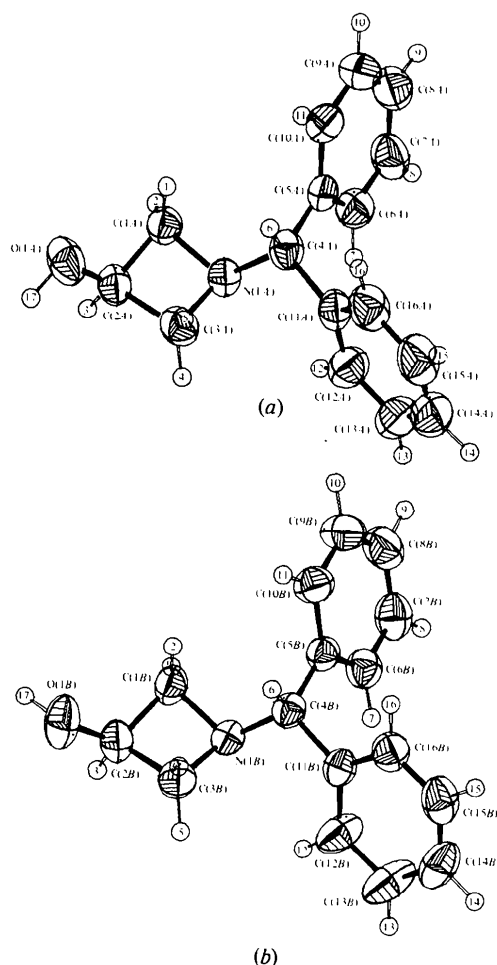


Fig. 2. Thermal ellipsoid plots for (a) molecule *A* and (b) molecule *B*.

### Conformation of the molecule

The four-membered rings are buckled, the angle between the planes  $N(1)-C(1)-C(3)$  and  $C(1)-C(2)-C(3)$  being  $156^\circ$  for molecule *A* and  $153^\circ$  for molecule *B* (Table 6). The dihedral angles for this ring are compared with the values in related compounds in Table 6. The four-membered ring tends to be planar when there is an exocyclic double bond (Karthä & Ambady, 1973; Colens, Declercq, Germain, Putzeys & Van Meerssche, 1974). According to Towns & Trefonas

Table 6. Details of the conformation of the four-membered ring in different structures

Compound	Angle of puckering ( $\theta$ )	Reference
(1) 1-Benzyl-1,2,2-trimethylazetidinium bromide	$176^\circ$	Moret & Trefonas (1968)
(2) 1-Benzyl-1,3,3-trimethylazetidinium iodide	$170$	Towns & Trefonas (1971)
(3) 1,1-Dibenzyl-3,3-dimethylazetidinium bromide	$180$	Snyder, McGandy, Van Etten, Trefonas & Towns (1969)
(4) <i>L</i> -Azetidine-2-carboxylic acid	$169$	Berman, McGandy, Burgner & Van Etten (1969)
(5) <i>N</i> -Methyl- <i>N</i> - <i>tert</i> -butyl-3-hydroxyazetidinium methanesulphonate	$166$	McGandy, Berman, Burgner & Van Etten (1969)
(6) 1-(Diphenylmethyl)azetidin-3-ol, Molecule <i>A</i>	$156$	Present study
Molecule <i>B</i>	$153$	Present study
(7) 1-(1-Methyl-2-phenylethyl)-2-methyl-3-hydroxyazetidinium hydrochloride	$154$	Wetherington & Moncrief (1974)

Table 7. *Some torsion angles*

	Molecule A	Molecule B
N(1)–C(4)–C(5)–C(10)	114.7°	104.4°
N(1)–C(4)–C(11)–C(16)	–142.6	–165.8

(1971) the four-membered azetidine ring is buckled to an extent of about  $10^\circ$  ( $\theta = 170^\circ$ ). The larger buckling in (5) (Table 6) is probably due to the O–H group which is involved in hydrogen bonding. The buckling is much larger in the present case as well as in (7) (Table 3) where both the O–H group and the N are involved in hydrogen bonding. The cyclobutane ring is either planar, or non-planar with a dihedral angle of  $26 + 3^\circ$  ( $\theta = 154 \pm 3^\circ$ ). Only molecules which possess a centre of inversion have a planar cyclobutane ring. There is no cyclobutane ring having a dihedral angle in the range  $1-18^\circ$  ( $\theta = 179-162^\circ$ ). On the other hand, the azetidine ring is found to be puckered with  $\theta$  ranging from  $153$  to  $180^\circ$ . O(1) and C(4) are *cis* with respect to each other and are substituted on the convex side of the azetidine ring. The buckling of the ring is so as to minimize the intramolecular steric interactions between O(1) and N(1) as well as between C(2) and C(4). The conformations of the four-membered ring of the two independent molecules are similar. However, the conformations of the phenyl rings with respect to the four-membered rings are quite different (Table 7), the angles being N(1A)–C(4A)–C(5A)–C(10A)  $114.7(5)$ ; N(1A)–C(4A)–C(11A)–C(16A)  $-142.6(5)$ , N(1B)–C(4B)–C(5B)–C(10B)  $104.4(5)$  and N(1B)–C(4B)–C(11B)–C(16B)  $-165.8(4)^\circ$ . This difference in conformation is presumably due to the different environments of the independent molecules in the lattice. The different conformations of the molecule may imply that the energy of the molecule has a double minimum or a shallow minimum with respect to rotation of the phenyl rings about C(4)–C(5) and C(4)–C(11).

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