There is a noticeable shortening effect for one bond in each ring, *i.e.* C(7)—C(8) 1·370 (6) and C(1)—C(6) 1·366 (7) Å; no explanation can be offered for this, but exactly the same effect was observed in the 2-chloro derivative (Shefter, 1976) where the two bonds are 1·376 and 1·361 Å, respectively. S—C(7) at 1·775 (4) Å agrees with 1·766 Å found in sodium naphthionate tetrahydrate (Brown & Corbridge, 1966) and S—N(1) at 1·661 (3) Å agrees with 1·666 Å in γ-sulphanilamide (Alléaume & Decap, 1965). The other bonds in the molecule are similar to those found in other sulphanilamides and sulphadiazines; a table of comparisons is given by Cook & Turner (1975).

The two rings are effectively planar, for C(1)–(6) the r.m.s. deviation is 0·030 (5) Å and for C(7)–(12) 0·012 (4) Å. The dihedral angle between them is 50·65 (5)°.

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Structure of 1-Diphenylmethyl-3-hydroxyazetidinium Chloride,* $C_{16}H_{18}NO^+.Cl^-$

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Abstract. $M_r = 275·8$, monoclinic, $P2_1/a$, $a = 12·356\ (5)$, $b = 9·054\ (4)$, $c = 14·043\ (4)$ Å, $\beta = 100·34\ (3)°$, $V = 1545·5$ Å^3, $Z = 4$, $D_m = 1·14$, $D_x = 1·185$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0·7107$ Å) $= 2·77$ mm^{-1}, $F(000) = 584·0$, $T = 293$ K, $R = 0·053$ for 1088 reflections. The four-membered ring is buckled 13·0° ($\theta = 167·0°$). The azetidinium moiety is linked to the Cl$^-$ ion through a hydrogen bond [O—H···Cl = 3·166 (5) Å].

Introduction. Structural studies on compounds containing the azetidine ring are important for the interpretation of their stereochemical and biological significance. In this connection our findings on the molecular geometry of the title compound (DPHA) are presented. The samples of DPHA were provided by Dr Joseph John of the Department of Science and Technology, India.

Experimental. Small, pale-yellow needles (from acetone solution). D_m by flotation in bromoform/benzene. Crystal 0·38 × 0·25 × 0·5 mm. Enraf–Nonius CAD-4/11M single-crystal X-ray diffractometer. Lattice parameters from 20 reflections (12° < 2θ < 26°). Data collection: h, k, $\pm l$ with $2\theta \leq 48°$; Mo $K\alpha$ radiation, graphite monochromator; $\omega/2\theta$ scan mode. Three standard reflections every 2000 s: no significant variation. 2707 independent reflections, 1088 with $F > 3\sigma(F)$. Lp correction. No absorption or decay corrections. $P2_1/a$, general positions $\pm(x, y, z; \frac{1}{2}-x, \frac{1}{2}+y, -z)$, from systematic absences $0k0$, $k \neq 2n$ and $h0l$, $h \neq 2n$. Direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Anisotropic full-matrix refinement for non-H atoms (*LALS*; Gantzel, Sparks & Trueblood, 1961). H from stereochemical considerations and verified from ΔF synthesis, only positions refined, $R = 0·053$, $R_w = 0·057$; $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/(a + bF + cF^2)$ with $a = 3·5$, $b = 1·0$ and $c = 0·025$. $(\Delta/\sigma)_{\max} = 0·01$. Final $\Delta\rho$ map featureless. No correction for

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secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non*-H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}(Å^2)
Cl	1102 (1)	4120 (2)	8744 (1)	3·7 (1)
O(1)	333 (4)	2088 (6)	10332 (3)	4·6 (2)
N(1)	−1786 (4)	2502 (6)	8567 (4)	2·5 (2)
C(1)	−1473 (5)	3157 (7)	9580 (5)	2·7 (2)
C(2)	−773 (6)	1791 (8)	9916 (5)	3·3 (2)
C(3)	−923 (6)	1252 (8)	8862 (5)	2·8 (3)
C(4)	−1682 (5)	3413 (7)	7724 (5)	3·1 (2)
C(5)	−2499 (5)	4678 (7)	7637 (4)	2·5 (2)
C(6)	−2098 (6)	6066 (9)	7920 (5)	3·8 (2)
C(7)	−2811 (8)	7261 (9)	7854 (6)	5·3 (4)
C(8)	−3923 (8)	7056 (9)	7531 (6)	5·7 (4)
C(9)	−4309 (6)	5692 (10)	7250 (6)	3·9 (3)
C(10)	−3607 (5)	4494 (7)	7319 (5)	3·1 (2)
C(11)	−1760 (5)	2450 (7)	6815 (4)	3·0 (2)
C(12)	−1043 (5)	2812 (7)	6195 (4)	4·8 (3)
C(13)	−1066 (5)	1964 (8)	5346 (5)	6·2 (3)
C(14)	−1794 (5)	855 (8)	5149 (5)	6·6 (3)
C(15)	−2687 (5)	515 (8)	5765 (5)	6·5 (3)
C(16)	−2470 (5)	1273 (7)	6623 (5)	4·8 (3)

Table 2. *Bond distances* (Å) *and angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

O(1)–C(2)	1·413 (8)	N(1)–C(4)	1·468 (8)
N(1)–C(3)	1·559 (9)	N(1)–C(1)	1·525 (8)
C(5)–C(6)	1·383 (9)	C(5)–C(10)	1·372 (9)
C(5)–C(4)	1·517 (9)	C(6)–C(7)	1·39 (1)
C(8)–C(7)	1·38 (1)	C(8)–C(9)	1·36 (1)
C(9)–C(10)	1·38 (1)	C(4)–C(11)	1·535 (9)
C(11)–C(12)	1·388 (8)	C(11)–C(16)	1·375 (9)
C(12)–C(13)	1·415 (9)	C(13)–C(14)	1·343 (9)
C(14)–C(15)	1·550 (9)	C(15)–C(16)	1·371 (9)
C(3)–C(2)	1·537 (9)	C(1)–C(2)	1·535 (9)
C(4)–N(1)–C(3)	117·8 (5)	C(4)–N(1)–C(1)	119·3 (5)
C(3)–N(1)–C(1)	89·3 (4)	C(6)–C(5)–C(10)	119·3 (6)
C(6)–C(5)–C(4)	117·6 (6)	C(10)–C(5)–C(4)	123·1 (6)
C(5)–C(6)–C(7)	119·9 (7)	C(6)–C(7)–C(8)	119·9 (8)
C(7)–C(8)–C(9)	119·7 (8)	C(8)–C(9)–C(10)	120·7 (7)
C(5)–C(10)–C(9)	120·3 (6)	N(1)–C(4)–C(5)	109·6 (5)
N(1)–C(4)–C(11)	110·5 (5)	C(5)–C(4)–C(11)	114·8 (5)
C(4)–C(11)–C(12)	115·8 (5)	C(4)–C(11)–C(16)	123·1 (6)
C(12)–C(11)–C(16)	121·1 (6)	C(11)–C(12)–C(13)	118·7 (6)
C(12)–C(13)–C(14)	119·5 (6)	C(13)–C(14)–C(15)	123·4 (6)
C(14)–C(15)–C(16)	110·2 (6)	C(11)–C(16)–C(15)	125·3 (6)
N(1)–C(3)–C(2)	89·1 (5)	N(1)–C(1)–C(2)	90·4 (5)
O(1)–C(2)–C(3)	113·5 (5)	O(1)–C(2)–C(1)	115·1 (5)
C(3)–C(2)–C(1)	89·7 (5)		

Table 3. *Some important torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)–N(1)–C(3)–C(2)	9·0 (5)	C(4)–N(1)–C(3)–C(2)	132·1 (5)
C(3)–N(1)–C(1)–C(2)	−9·0 (5)	C(4)–N(1)–C(1)–C(2)	−130·9 (5)
N(1)–C(3)–C(2)–C(1)	−8·9 (5)	N(1)–C(3)–C(2)–O(1)	−126·3 (4)
N(1)–C(1)–C(2)–C(3)·	9·1 (5)	N(1)–C(1)–C(2)–O(1)	125·1 (5)

Discussion. Atomic coordinates are given in Table 1.* Bond lengths and bond angles involving non-H atoms are given in Table 2. Table 3 gives some important torsion angles. Fig. 1 gives an *ORTEP* plot (Johnson, 1965) of the molecule.

The endocyclic N—C bonds [1·559 (9) and 1·525 (8) Å] are significantly longer than the exocyclic one which is 1·468 (8) Å. This feature is generally found in similar structures, such as 1-diphenylmethylazetidin-3-ol (hereafter DPAO) (Ramakumar, Venkatesan & Rao, 1977) and 3-hydroxy-2-methyl-1-(1-methyl-2-phenylethyl)azetidinium chloride (hereafter MPHA) (Wetherington & Moncrief, 1974). However, these bonds are particularly sensitive to the nature of the hydrogen-bond formation. In DPAO, the endocyclic bonds average 1·496 (7) Å while in MPHA these are 1·546 (15) and 1·457 (15) Å. DPHA has a hydrogen bond of the type O—H···Cl [O(1)···Cl = 3·166 (5), O(1)—H(O1) = 1·01 (5), Cl···H(O1) = 2·19 (6) Å, O(1)—H(O1)···Cl = 166·5°], while DPAO has a hydrogen bond of the type O—H···N [2·815 (7) Å] and in MPHA there are two hydrogen bonds of the types O—H···Cl [3·083 (11) Å] and N—H···Cl [3·095 (9) Å].

The angles within the four-membered ring in DPHA are close to 90° – in DPAO and MPHA one of the angles deviates significantly [86·4 (4) and 86·9 (8)° respectively].

The four-membered ring is buckled. The angle between the planes N(1),C(1),C(3) and C(1),C(2),C(3) is 13° (the angle of puckering, $\theta = 167{\cdot}0°$). This compares well with the structures of L-azetidine-2-carboxylic acid ($\theta = 169{\cdot}0°$; Berman, McGandy, Burgner & Van Etten, 1969), *N-tert*-butyl-3-hydroxy-*N*-methylazetidinium methanesulphonate ($\theta = 166{\cdot}0°$; McGandy, Berman, Burgner & Van Etten, 1969) and falls within the predicted values of Towns & Trefonas (1971). However, DPAO and MPHA differ significantly ($\theta = 154{\cdot}0°$ in both cases) and this can be attributed

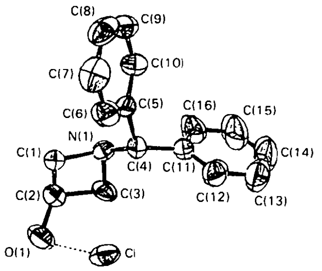

Fig. 1. A perspective view of the molecule.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39116 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to the involvement of the ring N in hydrogen-bond formation. The two phenyl rings are inclined to one another at an angle of 67·2°.

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Structure of 8-Chloro-1-[(dimethylamino)methyl]-6-phenyl-4*H*-imidazo-[1,2-*a*][1,4]benzodiazepine, $C_{20}H_{19}ClN_4$*

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Abstract. $M_r = 350·8$, triclinic, $P\bar{1}$, $a = 9·036$ (4), $b = 9·888$ (3), $c = 11·035$ (7) Å, $\alpha = 94·62$ (4), $\beta = 100·40$ (5), $\gamma = 106·75$ (3)°, $U = 919·4$ Å^3, $Z = 2$, $D_x = 1·267$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0·71069$ Å) $= 0·18$ mm^{-1}, $F(000) = 368$, room temperature, $R = 0·042$ for 1361 observed reflections. The seven-membered ring adopts a boat conformation and the angle between the phenyl ring and the fused benzene moiety is 66·5 (5)°. Bond lengths and angles agree with those found in other 1,4-benzodiazepines. The benzo and imidazo rings are effectively planar.

Introduction. Many 5-phenyl-1,4-benzodiazepines exhibit a broad range of psychotherapeutic properties. The title compound (Gall & Kamdar, 1981) is of the type possessing a five-membered hetero-ring fused across the N(1)—C(2) bond of the parent system. It has a rather low affinity for the benzodiazepine receptor *in vitro* in comparison with the analogous triazolobenzodiazepines triazolam† (Hester, Rudzik & Kamdar, 1971) and estazolam‡ (Meguro & Kuwada, 1970) which bind considerably better, approximately 100 times more strongly in the case of estazolam (Braestrup & Squires, 1978). The structure of the title compound is reported as part of a study of structure–activity relationships for benzodiazepines.

Experimental. Crystal size 0·025 × 0·15 × 0·5 mm, Enraf–Nonius CAD-4 diffractometer, cell dimensions from setting angles of 25 reflections, graphite-monochromated Mo $K\alpha$ radiation, no absorption correction, 3237 reflections scanned by ω–2θ scans up to $\theta = 25°$, 1361 reflections considered observed [$I > 2·5\sigma(I)$], index range h −10 to 9, k −11 to 11, l 0 to 12. Two standard reflections, measured every 2 h; no significant intensity variation. Structure solved by direct methods; H atoms apart from those of methyl groups located from Fourier difference map; least-squares refinement, $\sum w(\Delta F)^2$ minimized, methyl groups refined as rigid groups, other H atoms refined isotropically and non-H anisotropically; final calculated shifts all $<0·1\sigma$; $R = 0·042$, $wR = 0·052$; weighting scheme, $w = 1/[\sigma^2(F) + 0·0015F^2]$; residual electron density in final difference map within $\pm 0·2$ e Å^{-3}; no correction for secondary extinction.

Computations were carried out with *SHELX* (Sheldrick, 1978) using complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974) and *PLUTO* (Motherwell & Clegg, 1978).

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* Contribution from the Crystallography Unit, Universities of Aston and Birmingham.

† 8-Chloro-6-(2-chlorophenyl)-1-methyl-4*H*-*s*-triazolo[4,3-*a*][1,4]benzodiazepine. Marketed as Halcion (Upjohn).

‡ 8-Chloro-6-phenyl-4*H*-*s*-triazolo[4,3-*a*][1,4]benzodiazepine.

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