−175·2 and −171·7° in molecules 1 and 2, respectively. The fact that the conformations of the energy-minimized structures are at one edge of the distribution indicates that either the distribution in the solid state is systematically skewed by packing interactions or the calculations are not accurately simulating intramolecular interaction. Since no characteristic packing interaction or patterns are discernable in the 27 structures in the sample it seems more plausible to suggest that the calculations are limited. Analysis of crystallographic observation of the progesterone side chain *versus* molecular-mechanics calculations indicates that H—C—C=O and C—C—C=O parameters in *MM2p* are not adequately defined (Duax, Griffin & Rohrer, 1981). This could account for the results observed here.

This research was supported in part by NIAAMDD Grant No. DK26546 (formerly AM-26546) and DDR Grant No. RR-05716 and by RP.II.10 from the Polish Ministry of Science and Higher Education.

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Acta Cryst. (1988). C**44**, 1433–1435

Structure of an Antihistaminic Drug, Thonzylamine Hydrochloride

BY MASOOD PARVEZ

Department of Chemistry, The Pennsylvania State University, University Park, PA 16802, *USA*

(*Received* 5 *October* 1987; *accepted* 17 *March* 1988)

Abstract. *N*-[(4-Methoxyphenyl)methyl]-*N'*,*N'*-dimethyl-*N*-2-pyrimidinyl-1,2-ethanediamine hydrochloride, $C_{16}H_{23}N_4O^+.Cl^-$, $M_r = 322{\cdot}84$, orthorhombic, $Pca2_1$, $a = 9{\cdot}916\,(3)$, $b = 15{\cdot}722\,(3)$, $c = 10{\cdot}840\,(8)$ Å, $V = 1690\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}231$ mm^{-1}, $F(000) = 688$, $T = 293\,(1)$ K, $R = 0{\cdot}042$ for 2218 observed data with $I > 3\sigma(I)$. Both six-membered rings are individually planar and the configurations at bonds N(1)—C(9) and C(5)—C(6) are *cis* and *trans*, respectively. The cation is protonated at the terminal dimethylamino group and is hydrogen bonded to the Cl^- ion, N···Cl 3·031 (3) Å.

Introduction. Thonzylamine is an active component of some cold remedies (Witiak, 1970). Unlike tripelennamine, which is a potent antihistaminic drug widely used as a cure for hay fever and other allergies, thonzylamine has a lower toxicity and causes markedly less drowsiness than the former (Wilhelm, 1961). The crystal structure of thonzylamine hydrochloride has been determined to establish its conformation as part of a program correlating biological activity and molecular structures of antihistamine drugs containing an alkyldiamine group.

Experimental. Colorless crystals of thonzylamine hydrochloride (Sigma Inc.) were grown from methanol by slow evaporation at room temperature. A crystal of approximate dimensions 0·45 × 0·32 × 0·70 mm was obtained by cutting a large plate and used for data collection. Cell dimensions were determined by a least-squares refinement of the setting angles of 25 reflections with θ in the range 10–17°. Intensity data in the range $2 < \theta < 30°$ were collected on an Enraf–Nonius CAD-4 diffractometer using the $\omega/2\theta$ scan technique, variable scan speed (0·72–2·35° min^{-1}) and monochromatized Mo $K\alpha$ radiation. The intensities of three standard reflections, monitored at intervals of 2 h exposure time, decreased by 4·8% (linear decay) over the period of the data collection; this decay was corrected for by appropriate scaling. 2990 intensity maxima were collected (h 0→13, k −22→0, l 0→15) and corrected for Lorentz, polarization and absorption effects (North, Phillips & Mathews, 1968); the maximum and minimum transmission factors were

0·996 and 0·978, respectively. Of the 2578 unique reflections, 2218 with $I > 3\sigma(I)$ were labeled observed and used in all subsequent calculations.

The structure was solved by the heavy-atom method. The coordinates of the Cl^- ion were obtained from a three-dimensional Patterson synthesis and those of the non-H atoms from a subsequent Fourier map. Refinement by full-matrix least-squares calculations on F's using unit weights initially with isotropic thermal parameters lowered R to 0·062. A difference map calculated at this stage showed electron-density maxima 0·3–0·8 e Å^{-3} in positions expected for the H atoms. H atoms were included in the refinement at geometrically idealized positions (C—H and N—H 0·95 Å) with an overall isotropic temperature factor $B_{\rm iso} = 5{\cdot}0$ Å^2. The refinement converged completely with $R = 0{\cdot}042$ and $wR = 0{\cdot}061$, where $w = [\sigma^2(F_o) + (0{\cdot}01\,F_o)^2]^{-1}$; the maximum shift/e.s.d. in the last cycle of refinement being $< 0{\cdot}01$ and goodness of fit, $S = 1{\cdot}114$. A final difference map was devoid of significant features with $\Delta\rho$ in the range −0·23 to 0·28 e Å^{-3}. Scattering factors used in the calculations were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) and allowance was made for anomalous dispersion (Cromer & Liberman, 1970). The absolute structure could not be determined from the data. Computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985) and *ORTEP*II (Johnson, 1976).

Discussion. Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are listed in Table 1.* Table 2 contains bond lengths, angles and important torsion angles. Fig. 1 shows the molecular structure of thonzylamine hydrochloride with the crystallographic numbering scheme. The diaminoethyl chain is fully extended and the plane of the side chain comprising N(1), C(5), C(6), N(4), C(8) is inclined at 82·2 (1) and 36·6 (2)° to the planes of the pyrimidine and phenyl rings, respectively. The pyrimidine and the phenyl rings are inclined at 63·5 (1)° and both the rings are individually planar with maximum displacements of atoms being 0·009 (2) and 0·010 (3) Å, respectively. The molecule adopts the *trans* configuration about the C(5)—C(6) bond and the *cis* configuration about N(1)—C(9) bond. In the pyrimidine ring, the C—N distances are in excellent agreement [range 1·328–1·343 (3) Å, mean 1·337 (3) Å] while the agreement in the C—C distances, C(2)—C(3) 1·366 (5) and C(3)—C(6) 1·348 (5) Å, is within 4σ limits. The aromatic ring

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{\rm eq}$
Cl	0·47112 (5)	0·07640 (4)	1·0259*	3·819 (9)
O	0·0886 (2)	0·5922 (1)	0·7744 (2)	4·70 (4)
N(1)	0·3923 (2)	0·2485 (1)	0·6212 (2)	2·92 (3)
N(2)	0·5362 (2)	0·2906 (1)	0·4648 (2)	3·96 (4)
N(3)	0·6144 (2)	0·2064 (1)	0·6339 (2)	3·97 (4)
N(4)	0·2734 (2)	0·0631 (1)	0·8135 (2)	2·81 (3)
C(1)	0·5189 (2)	0·2487 (1)	0·5712 (2)	2·80 (3)
C(2)	0·6596 (3)	0·2880 (2)	0·4173 (3)	4·74 (5)
C(3)	0·7632 (3)	0·2450 (2)	0·4723 (4)	5·40 (6)
C(4)	0·7359 (3)	0·2058 (2)	0·5801 (4)	5·01 (6)
C(5)	0·3659 (2)	0·2020 (1)	0·7350 (2)	2·90 (3)
C(6)	0·3316 (2)	0·1096 (1)	0·7057 (2)	2·76 (3)
C(7)	0·1415 (2)	0·0984 (2)	0·8540 (2)	3·63 (4)
C(8)	0·2576 (3)	−0·0287 (1)	0·7823 (3)	4·42 (5)
C(9)	0·2769 (2)	0·2853 (1)	0·5577 (2)	3·05 (3)
C(10)	0·2247 (2)	0·3663 (1)	0·6173 (2)	2·82 (3)
C(11)	0·2800 (2)	0·4449 (1)	0·5872 (2)	3·68 (4)
C(12)	0·2319 (3)	0·5191 (1)	0·6403 (3)	4·15 (5)
C(13)	0·1282 (2)	0·5157 (1)	0·7267 (2)	3·37 (4)
C(14)	0·0717 (2)	0·4384 (1)	0·7562 (2)	3·42 (4)
C(15)	0·1201 (2)	0·3640 (1)	0·7002 (2)	3·36 (4)
C(16)	−0·0148 (3)	0·5916 (2)	0·8643 (3)	4·52 (5)

* Fixed coordinate to define the origin.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

O—C(13)	1·367 (3)	N(4)—C(8)	1·490 (3)
O—C(16)	1·414 (4)	C(2)—C(3)	1·366 (5)
N(1)—C(1)	1·367 (3)	C(3)—C(4)	1·348 (5)
N(1)—C(5)	1·457 (3)	C(5)—C(6)	1·526 (3)
N(1)—C(9)	1·455 (3)	C(9)—C(10)	1·518 (3)
N(2)—C(1)	1·339 (3)	C(10)—C(11)	1·391 (3)
N(2)—C(2)	1·328 (3)	C(10)—C(15)	1·373 (3)
N(3)—C(1)	1·343 (3)	C(11)—C(12)	1·385 (4)
N(3)—C(4)	1·339 (4)	C(12)—C(13)	1·391 (4)
N(1)—C(6)	1·495 (3)	C(13)—C(14)	1·376 (3)
N(4)—C(7)	1·487 (3)	C(14)—C(15)	1·402 (3)

N(1)—C(1)—N(3)	116·5 (2)	O—C(13)—C(14)	124·9 (2)
N(2)—C(1)—N(3)	126·1 (2)	C(12)—C(13)—C(14)	119·4 (2)
N(2)—C(2)—C(3)	122·6 (3)	C(13)—C(14)—C(15)	119·8 (2)
C(2)—C(3)—C(4)	117·0 (3)	C(10)—C(15)—C(14)	121·4 (2)
N(3)—C(4)—C(3)	123·7 (3)	C(13)—O—C(16)	117·6 (2)
N(1)—C(5)—C(6)	110·0 (2)	C(1)—N(1)—C(5)	120·1 (2)
N(4)—C(6)—C(5)	112·9 (2)	C(1)—N(1)—C(9)	122·2 (2)
N(1)—C(9)—C(10)	113·6 (2)	C(5)—N(1)—C(9)	117·3 (2)
C(9)—C(10)—C(11)	120·7 (2)	C(1)—N(2)—C(2)	115·9 (2)
C(9)—C(10)—C(15)	121·0 (2)	C(1)—N(3)—C(4)	114·7 (2)
C(11)—C(10)—C(15)	118·3 (2)	C(6)—N(4)—C(7)	112·8 (2)
C(10)—C(11)—C(12)	121·0 (2)	C(6)—N(4)—C(8)	109·7 (2)
C(11)—C(12)—C(13)	120·1 (2)	C(7)—N(4)—C(8)	109·6 (2)
O—C(13)—C(12)	115·7 (2)	N(1)—C(1)—N(2)	117·4 (2)

C(16)—O—C(13)—C(14)	2·6 (3)	C(8)—N(4)—C(6)—C(5)	−173·5 (3)
C(9)—N(1)—C(1)—N(2)	5·4 (3)	N(1)—C(5)—C(6)—N(4)	−167·0 (4)
C(1)—N(1)—C(5)—C(6)	−86·7 (3)	N(1)—C(9)—C(10)—C(11)	86·3 (4)
C(1)—N(1)—C(9)—C(10)	−110·1 (3)	C(15)—C(10)—C(11)—C(12)	0·6 (4)
C(5)—N(1)—C(9)—C(10)	76·9 (3)	C(11)—C(10)—C(15)—C(14)	−1·6 (4)
C(2)—N(2)—C(1)—N(3)	1·2 (3)	C(10)—C(11)—C(12)—C(13)	1·1 (4)
C(1)—N(2)—C(2)—C(3)	0·4 (3)	C(11)—C(12)—C(13)—C(14)	−1·7 (4)
N(2)—C(2)—C(3)—C(4)	−1·3 (4)	C(12)—C(13)—C(14)—C(15)	0·7 (4)
C(2)—C(3)—C(4)—N(3)	0·7 (4)	C(13)—C(14)—C(15)—C(10)	1·0 (4)
C(4)—N(3)—C(1)—N(2)	−1·7 (3)		
C(1)—N(3)—C(4)—C(3)	0·7 (3)		

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* Lists of structure factors, anisotropic temperature factors, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44872 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

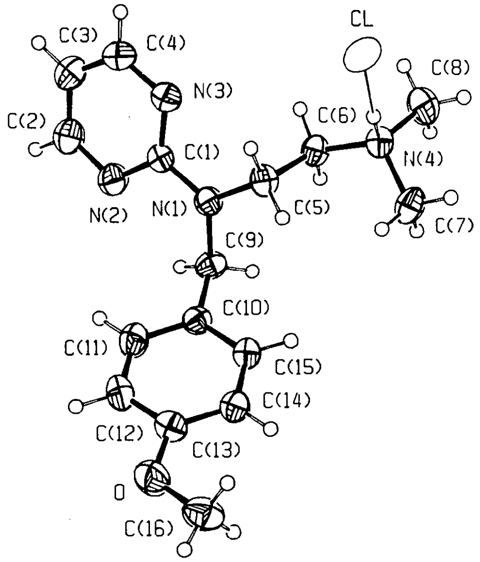

Fig. 1. An *ORTEP* (Johnson, 1976) view of thonzylamine hydrochloride with crystallographic numbering scheme.

C—C distances range between 1·373 (3) and 1·402 (3) Å [mean 1·386 (3) Å]. The N(1)—C(5) and N(1)—C(9) distances [1·457 and 1·455 (3) Å, respectively] are equal but significantly shorter than the N—C distances involving the N(4) atom which range between 1·487–1·495 (3) Å [mean 1·491 (3) Å]. A comparison of the angles around N(1) [120·1 (2), 122·2 (2) and 117·3 (2)°] and N(4) [112·8 (2), 109·7 (2) and 109·6 (2)°] clearly suggests that the two N atoms are different from each other and that N(1) is sp^2 hybridized while N(4) is sp^3 hybridized. Similar structural features have been reported in the structures of 2-{*N*-[2-(dimethylamino)ethyl]-2-thenylamino}pyridine hydrochloride (Clark & Palenik, 1972) and tripelennamine hydrochloride (Parvez, 1987). The rest of the molecular dimensions are in good agreement with the values found in other organic structures.

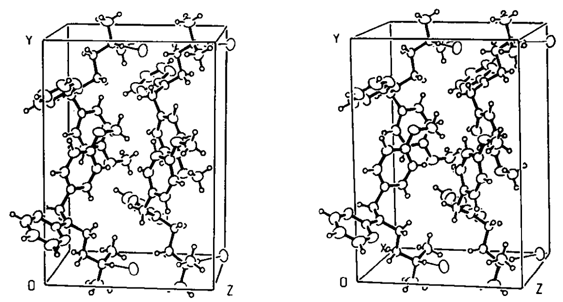

Fig. 2. A stereoview of the unit cell showing molecular packing.

The crystal structure consists of the cation containing an H atom on the dimethylamino group [H(N4)] which forms a relatively strong hydrogen bond to the anion, Cl⁻. The distances N(4)···Cl and H(N4)···Cl are 3·031 (2) and 2·08 Å, respectively, and the Cl···H(N4)—N(4) angle is 176·8°. There are no unusual intermolecular distances less than van der Waals distances and the crystal is composed of hydrogen-bonded cation–anion species (Fig. 2).

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Acta Cryst. (1988). C**44**, 1435–1438

1-Phosphabicyclo[3.3.1]nonane 1-Sulfide

By U. Baumeister, H. Hartung and F. Krech

Sektion Chemie, Martin-Luther-Universität Halle-Wittenberg, Mühlpforte 1, *DDR*-4020 *Halle, German Democratic Republic*

(*Received* 30 *October* 1987; *accepted* 7 *April* 1988)

Abstract. $C_8H_{15}PS$, $M_r = 174{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}270\ (1)$, $b = 12{\cdot}350\ (3)$, $c = 10{\cdot}275\ (2)$ Å, $\beta = 98{\cdot}60\ (1)°$, $V = 912{\cdot}1\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}27$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 0·403 mm^{-1}, $F(000) = 376$, $T = 293$ K, final $R = 0{\cdot}032$ for 1163 observed reflections. The title compound adopts a chair–chair conformation in the solid state with the central three-atom plane as a (non-

0108-2701/88/081435-04$03.00