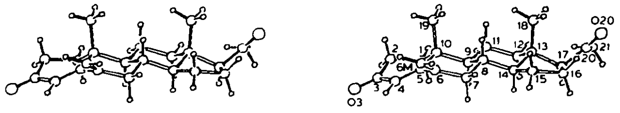

Fig. 1. Stereoview of the molecule with the atomic numbering scheme.

Atomic positional and equivalent displacement parameters for non-H atoms are listed in Table 1. Bond lengths and valency angles are listed in Table 2.* A stereoscopic view (Johnson, 1965) of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1.

Related literature. This compound has been shown to be a good irreversible inhibitor of the steroidal enzyme 5α-reductase (Petrow, Wang, Lack & Sandberg, 1981). The overall conformation of the molecule is very similar to the conformation of 17α-acetoxy-6-methylene-4-pregnene-3,20-dione (Petrow, Padilla, McPhail, Bruchovsky & Schneider, 1989), another potent 5α-reductase inhibitor and to progesterone itself (Campsteyn, Dupont & Dideberg, 1972).

Research supported in part by RP.II.10 grant from the Polish Ministry of National Education (to ZG, PG and ZW) and by NIAAMD Grant No. DK26546 (formerly AM-26546) and DDR Grant No. RR-05716 (to WLD and PDS).

---

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52514 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Blessing, R. H. (1987). *Crystallogr. Rev.* **1**, 3–58.

Campsteyn, H., Dupont, L. & Dideberg, O. (1972). *Acta Cryst.* **B28**, 3032–3036.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Petrow, V., Padilla, G. M., McPhail, A. T., Bruchovsky, N. & Schneider, S. L. (1989), *J. Steroid Biochem.* **32**, 399–407.

Petrow, V., Wang, Y., Lack, L. & Sandberg, A. (1981). *Steroids*, **38**, 121–140.

Acta Cryst. (1990). **C46**, 943–945

Structure of an Anhistaminic Drug, Racemic Chlorpheniramine Hydrogen Maleate

By Masood Parvez

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

(*Received* 2 *October* 1989; *accepted* 11 *December* 1989)

Abstract. γ-(4-Chlorophenyl)-*N*,*N*-dimethyl-2-pyridinepropanaminium hydrogen maleate, $C_{16}H_{20}ClN_2^+.C_4H_3O_4^-$, $M_r = 390{\cdot}87$, monoclinic, $P2_1/n$, $a = 9{\cdot}769\,(2)$, $b = 10{\cdot}757\,(3)$, $c = 19{\cdot}139\,(5)$ Å, $\beta = 90{\cdot}97\,(2)°$, $V = 2010{\cdot}9\,(15)$ Å^3, $Z = 4$, $D_x = 1{\cdot}291$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}213$ mm^{-1}, $F(000) = 824$, $T = 293\,(1)$ K, $R = 0{\cdot}050$ for 2276 observed data with $I > 3\sigma(I)$. The pyridine and the phenyl rings are inclined at an angle of 105·0° and the side chain comprising C(6), C(7), C(8), C(9) and N(2) atoms is fully extended. The maleate anion is hydrogen bonded to the dimethylamino group [O(4)···H(N2) 1·86 (2) Å, O(4)···H(N2)—N(2) 171 (2)°]. The maleate anion also shows a strong intramolecular hydrogen bond [O···H(O) 1·47 (2) Å, O—H(O)···O 174 (2)°].

Experimental. Thin plates of chlorpheniramine hydrogen maleate (Sigma, Inc.) were grown from ethanol solution by slow evaporation at room temperature. A crystal of approximate size 0·47 × 0·52 × 0·20 mm was cut from a large plate for data collection. Accurate cell constants and an orientation matrix were determined by a least-squares refinement of the setting angles of 25 reflections with θ in the range 10–15°. Intensity data in the range $2 < \theta < 25°$ (h $0 \rightarrow 11$, k $0 \rightarrow 12$, l $-22 \rightarrow 22$) were collected on an Enraf–Nonius CAD-4 diffractometer using the $\omega/2\theta$ scan method, variable scan speed (1·25–2·35° min^{-1}) and monochromatized Mo $K\alpha$ radiation. The intensities of three standard reflections measured every 2 h of exposure time decayed linearly 3·9% over the period of data collection; this decay was corrected

0108-2701/90/050943-03$03.00

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

$$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl	0·4210 (1)	0·1418 (1)	0·9517 (1)	8·38 (2)
O(1)	0·9614 (2)	0·3168 (2)	0·9391 (1)	5·57 (4)
O(2)	1·0439 (2)	0·2627 (2)	1·0412 (1)	8·04 (6)
O(3)	0·8209 (2)	0·2221 (2)	0·8488 (1)	6·11 (5)
O(4)	0·7170 (2)	0·0445 (2)	0·8310 (1)	6·02 (5)
N(1)	0·0293 (2)	0·0309 (3)	0·6397 (1)	6·10 (6)
N(2)	−0·1427 (2)	−0·3436 (2)	0·7916 (1)	4·10 (4)
C(1)	0·1344 (3)	−0·0446 (2)	0·6531 (1)	4·51 (5)
C(2)	0·2459 (3)	−0·0488 (3)	0·6108 (2)	5·74 (7)
C(3)	0·2520 (3)	0·0239 (3)	0·5532 (2)	6·49 (7)
C(4)	0·1456 (4)	0·1013 (3)	0·5386 (2)	6·85 (8)
C(5)	0·0390 (3)	0·1019 (3)	0·5829 (2)	7·14 (8)
C(6)	0·1304 (3)	−0·1291 (2)	0·7167 (1)	4·58 (6)
C(7)	−0·0121 (3)	−0·1772 (2)	0·7306 (1)	4·59 (5)
C(8)	−0·0097 (3)	−0·2811 (3)	0·7846 (1)	4·67 (6)
C(9)	−0·1334 (3)	−0·4577 (3)	0·8350 (2)	5·91 (7)
C(10)	−0·2523 (3)	−0·2612 (3)	0·8190 (2)	5·46 (6)
C(11)	0·1982 (2)	−0·0640 (2)	0·7795 (1)	4·29 (5)
C(12)	0·3229 (3)	−0·1019 (3)	0·8038 (2)	5·57 (7)
C(13)	0·3921 (3)	−0·0408 (3)	0·8569 (2)	5·86 (7)
C(14)	0·3335 (3)	0·0604 (3)	0·8867 (1)	5·14 (6)
C(15)	0·2049 (3)	0·0992 (2)	0·8650 (2)	5·54 (6)
C(16)	0·1388 (3)	0·0363 (3)	0·8108 (1)	4·97 (6)
C(17)	0·9696 (3)	0·2421 (3)	0·9914 (1)	4·76 (6)
C(18)	0·8859 (3)	0·1285 (3)	0·9913 (1)	5·08 (6)
C(19)	0·8105 (3)	0·0757 (3)	0·9417 (1)	5·23 (6)
C(20)	0·7807 (3)	0·1167 (2)	0·8690 (1)	4·46 (5)

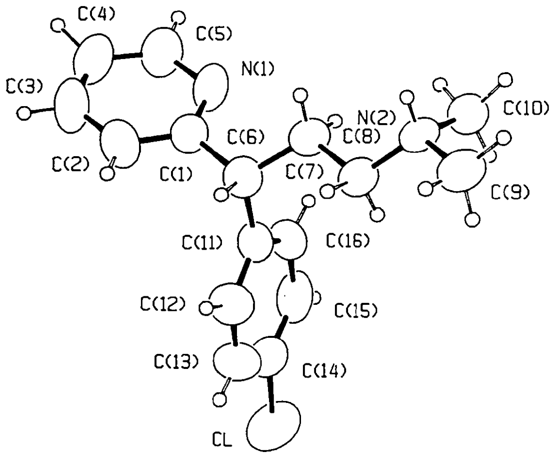

Fig. 1. The molecular structure of the chlorpheniramine cation with the crystallographic numbering scheme.

Table 2. *Bond distances* (Å) *and angles* (°)

Cl—C(14)	1·736 (3)	C(4)—C(5)	1·354 (5)
O(1)—C(17)	1·286 (3)	C(6)—C(7)	1·513 (4)
O(2)—C(17)	1·208 (3)	C(6)—C(11)	1·532 (4)
O(3)—C(20)	1·263 (3)	C(7)—C(8)	1·522 (4)
O(4)—C(20)	1·225 (3)	C(11)—C(12)	1·359 (4)
N(1)—C(1)	1·330 (4)	C(11)—C(16)	1·368 (4)
N(1)—C(5)	1·334 (5)	C(12)—C(13)	1·378 (4)
N(2)—C(8)	1·471 (3)	C(13)—C(14)	1·359 (5)
N(2)—C(9)	1·484 (3)	C(14)—C(15)	1·381 (5)
N(2)—C(10)	1·493 (3)	C(15)—C(16)	1·389 (4)
C(1)—C(2)	1·368 (4)	C(17)—C(18)	1·470 (4)
C(1)—C(6)	1·521 (4)	C(18)—C(19)	1·321 (4)
C(2)—C(3)	1·354 (4)	C(19)—C(20)	1·484 (4)
C(3)—C(4)	1·357 (5)		

C(1)—N(1)—C(5)	116·2 (2)	C(6)—C(11)—C(16)	121·6 (2)
C(8)—N(2)—C(9)	112·6 (2)	C(12)—C(11)—C(16)	118·1 (2)
C(8)—N(2)—C(10)	113·7 (2)	C(11)—C(12)—C(13)	122·4 (3)
C(9)—N(2)—C(10)	109·5 (3)	C(12)—C(13)—C(14)	119·1 (3)
N(1)—C(1)—C(2)	121·8 (2)	Cl—C(14)—C(13)	120·0 (2)
N(1)—C(1)—C(6)	119·3 (2)	Cl—C(14)—C(15)	119·8 (2)
C(2)—C(1)—C(6)	119·0 (2)	C(13)—C(14)—C(15)	120·2 (3)
C(1)—C(2)—C(3)	120·6 (3)	C(14)—C(15)—C(16)	119·1 (2)
C(2)—C(3)—C(4)	118·6 (3)	C(11)—C(16)—C(15)	121·1 (2)
C(3)—C(4)—C(5)	118·0 (3)	O(1)—C(17)—O(2)	121·9 (3)
N(1)—C(5)—C(4)	125·0 (3)	O(1)—C(17)—C(18)	119·4 (2)
C(1)—C(6)—C(7)	112·4 (3)	O(2)—C(17)—C(18)	118·7 (3)
C(1)—C(6)—C(11)	109·8 (3)	C(17)—C(18)—C(19)	131·4 (2)
C(7)—C(6)—C(11)	113·9 (2)	C(18)—C(19)—C(20)	130·2 (3)
C(6)—C(7)—C(9)	111·6 (3)	O(3)—C(20)—O(4)	123·0 (2)
N(2)—C(8)—C(7)	113·2 (2)	O(3)—C(20)—C(19)	119·8 (2)
C(6)—C(11)—C(12)	120·3 (2)	O(4)—C(20)—C(19)	117·2 (2)

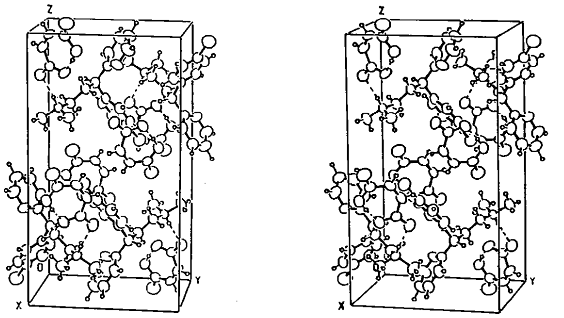

Fig. 2. A stereoview of the unit cell showing hydrogen bonds.

for by appropriate scaling. The data were corrected for Lorentz, polarization and absorption effects (North, Phillips & Mathews, 1968); the max. and min. transmission coefficients were 1·00 and 0·85, respectively. 4018 reflections measured, 3526 unique, 2276 with $I > 3\sigma(I)$ was labelled observed and used in all subsequent calculations.

The structure was solved by the heavy-atom method. The coordinates of the Cl atom were obtained from a three-dimensional Patterson map and those of the non-H atoms from a subsequent Fourier map. The structure was refined by full-matrix least-squares calculations on F's, initially with isotropic and finally with anisotropic temperature factors for the non-H atoms. A difference map calculated at an intermediate stage revealed the positions of H atoms which were included and allowed to refine with an overall fixed temperature factor B_{iso} = 5·0 Å^2. The refinement converged with R = 0·050 and wR = 0·071, where $w = [\sigma^2(F_o) + 0{\cdot}080(F_o)^2]^{-1}$; the maximum shift/e.s.d. in the last cycle of refinement was <0·03 and S = 1·538. A final difference map showed no significant features with $\Delta\rho$ in the range −0·26 to 0·32 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). 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).

Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are given in Table 1.* Table 2 contains bond lengths and bond angles. Fig. 1 shows the molecular structure of the chlorpheniramine cation with the crystallographic numbering scheme. Fig. 2 is a stereoview of the unit cell showing hydrogen bonds.

Related literature. The structure of (+)-chlorpheniramine maleate has been reported by James & Williams (1974).

References

B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

James, M. N. G. & Williams, G. J. B. (1974). *Can. J. Chem.* **52**, 1872–1879.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

---

* Lists of structure factors, anisotropic temperature factors, H-atom coordinates, molecular dimensions involving H atoms, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52530 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C**46**, 945–947

p-Nitrobenzenediazonium Tetrafluoroborate

By John C. Barnes

*Chemistry Department, The University, Dundee DD*1 4*HN*, *Scotland*

and Anthony Butler and Lesley Anderson

*Chemistry Department, University of St Andrews, St Andrews, KY*16 9*ST*, *Scotland*

(*Received* 27 *November* 1989; *accepted* 5 *January* 1990)

Abstract. $C_6H_4N_3O_2^+.BF_4^-$, $M_r = 236{\cdot}92$, orthorhombic, $Fdd2$, $a = 22{\cdot}56\,(5)$, $b = 31{\cdot}09\,(5)$, $c = 5{\cdot}33\,(3)$ Å, $V = 3738{\cdot}4$ Å^3, $Z = 16$, $D_x = 1{\cdot}68$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}26$ cm^{-1}, $F(000) = 1888$, $T = 293$ K, $R = 0{\cdot}081$ for 732 reflexions. The structure was determined because of an anomaly in the solid-state NMR spectrum of the title compound. The dimensions of the cation offer no evidence for a significant contribution from a quinonoid structure, $N{=}N^+$ 1·103 (8) Å. The normal to the mean plane of the nitro group lies at 11·4 (1)° to the normal to the mean plane of the benzene ring. The anion is disordered. Rotation about an F—B bond almost parallel to the *c* axis gives two sets of positions for the three F atoms. The site occupancy of the majority position is 82·3 (1·6)%. The shortest cation–anion contact is 2·621 (7) Å (N···F). There are several N···F and O···F distances in the range 3·1–3·3 Å.

Experimental. Off-white, striated needles were prepared from 4-nitroaniline by the method of Starky (1943) and recrystallized from acetonitrile by slow evaporation of the solvent at room temperature. Crystals deteriorated slowly on the open bench but showed no loss of intensity during data collection when mounted in Lindemann glass capillaries. Stoe Stadi-II diffractometer. Crystal 0·61 × 0·26 × 0·25 mm aligned along **c**. Unit cell refined from 20 precisely set reflexions with $12 < 2\theta < 15°$. 1418 measured reflexions gave 886 unique data ($R_{\mathrm{int}} = 0{\cdot}070$). Index range $-6 \le h \le 27$, $-2 \le k \le 37$, $0 \le l \le 5$, $1 \le 2\theta \le 50°$. 732 reflexions with $F_o > 2\sigma_F$ used in the refinement. Programs used: *SHELXS*86 (Sheldrick, 1986), *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *SHELX*76.

Structure solution by routine direct methods. Refinement minimizing $\sum w|F_o - |F_c||^2$ to $R = 0{\cdot}081$, $wR = 0{\cdot}095$. Difference map showed alternative positions for F(14), F(15), F(16) by rotation about F(13)—B(12), which is almost parallel to **c**. Majority position [82·3 (1·6%)] refined as rigid tetrahedron

0108-2701/90/050945-03$03.00