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Pargyline hydrochloride, a monoamine oxidase inhibitor: an orthorhombic form

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Key indicators

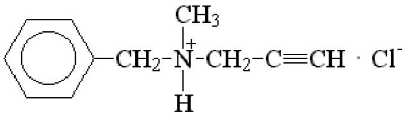
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.012 Å
R factor = 0.065
wR factor = 0.167
Data-to-parameter ratio = 7.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, *N*-benzyl-*N*-methylprop-2-yn-1-aminium chloride, $C_{11}H_{14}N^+\cdot Cl^-$, the asymmetric unit contains two independent enantiomeric cations with a pseudo-inversion center between them. In both molecules, the protonation occurs at the N atom, and the side chains are extended in a direction roughly perpendicular to the benzene rings. The molecules are arranged head-to-head and tail-to-tail, producing hydrophilic and hydrophobic regions. In addition to the ordinary N—H···Cl hydrogen bonds, numerous weak non-standard hydrogen bonds of the type C—H···Cl also contribute to the crystal packing.

Comment

Pargyline and chemically related compounds such as clorgyline and selegiline, which have an acetylenic grouppositioned β to an amino group, are irreversible inhibitors of flavin-linked monoamine oxidases (MAO) (Hellerman & Erwin, 1968). Inhibition occurs *via* covalent attachment of the acetylene to the flavin. Although structurally related to selegiline (deprenyl), which is used clinically in the treatment of Parkinson's disease, pargyline has a less pronounced selectivity towards MAO-B (Fuller, 1972). Nonetheless, pargyline has been used as an inhibitor in the crystal structure of MAO-B, where its position has been inferred in a 3.0 Å resolution electron-density map (Binda *et al.*, 2002). The crystal structure of pargyline may be useful in the interpretation of such protein-inhibitor complexes. The structure of pargyline hydrochloride, (I), in its orthrhombic form is presented in Fig. 1.



$C_6H_5\text{-}CH_2\text{-}\overset{CH_3}{\underset{H}{N^+}}\text{-}CH_2\text{-}C{\equiv}CH\cdot Cl^-$

(I)

Bond distances and angles are consistent with standard values. The asymmetric unit contains two independent enantiomeric molecules protonated at the N atom. Analysis of the positional parameters of the atoms implies the existence of a non-crystallographic center of symmetry between the two cations, located at (0.24, 0.40, 0.42). The benzene rings of the two independent cations are almost parallel, with an interplanar angle of 3.0 (5)°. The side chains extend roughly perpendicular to the benzene rings: the torsion angles C1—C7—N8—C10 are −53.0 (7) and 51.6 (7)° for cations *A* and *B*, respectively. In the crystal structure, the cations are packed in a head-to-head and tail-to-tail fashion, creating distinct

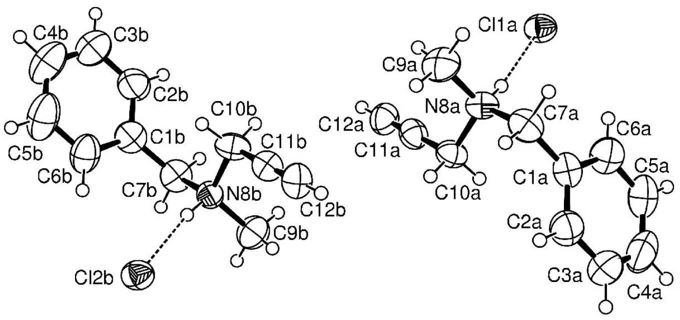


Figure 1
The structure of the asymmetric unit of pargyline hydrochloride, showing 50% probability displacement ellipsoids. H atoms are drawn as small circles of arbitrary radii. Hydrogen bonds are shown as dashed lines.

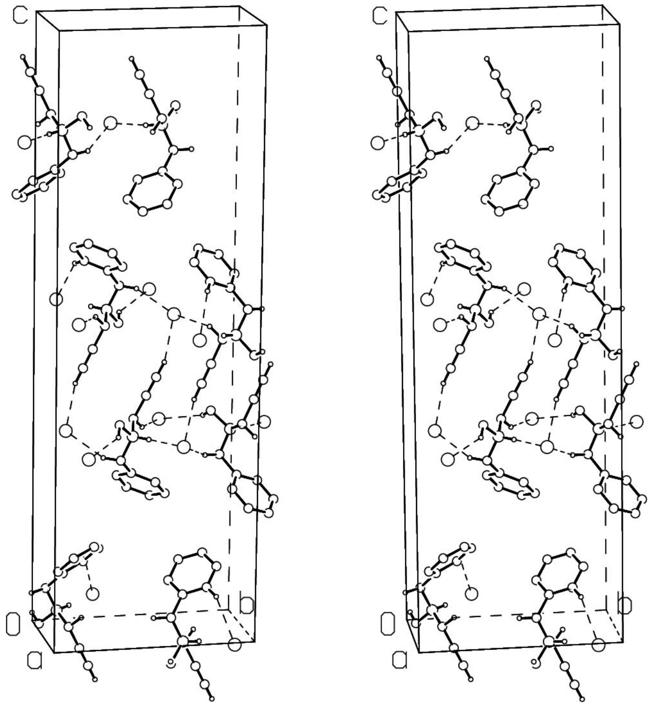


Figure 2
Stereoscopic diagram of the molecular packing and hydrogen-bond scheme (shown as dashed lines between atoms). Atoms are drawn as circles of arbitrary radii. For clarity, only the H atoms involved in hydrogen bonds are shown.

hydrophilic and hydrophobic regions which run perpendicular to the *c* axis (Fig. 2).

The only hydrogen-bond acceptors in this crystal structure are Cl^- anions. The two standard hydrogen bonds N—H···Cl, and a number of weak non-standard hydrogen bonds of the type C—H···Cl (Steiner, 1997), defining the coordination about the Cl ions (Table 1), contribute significantly to the molecular packing in the crystal. The usual van der Waals interactions are also present.

Experimental

To obtain crystals suitable for data collection, pargyline hydrochloride was dissolved in a chloroform–benzene mixture (1:1), and the solution subjected to slow evaporation at 278 K. Crystals appeared after two weeks. The crystals were small colorless needles. Attempts to grow better crystals proved unsuccessful.

Crystal data

$C_{11}H_{14}N^+\cdot Cl^-$
$M_r = 195.68$
Orthorhombic, $Pbc2_1$
$a = 5.958$ (2) Å
$b = 11.039$ (3) Å
$c = 33.596$ (5) Å
$V = 2209.6$ (10) Å^3
$Z = 8$
$D_x = 1.176$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 32 reflections
$\theta = 22–37°$
$\mu = 2.68$ mm^{-1}
$T = 294$ (2) K
Needle, colorless
0.31 × 0.07 × 0.03 mm

Data collection

Picker FACS-1 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.794$, $T_{\max} = 0.919$
1908 measured reflections
1908 independent reflections
1636 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 65.0°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 39$
3 standard reflections every 100 reflections intensity decay: 1.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.167$
$S = 0.98$
1908 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1152P)^2 + 0.9636P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0071 (9)
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter = 0.45 (8)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8*A*—H8*A*···Cl1*A*	0.91	2.13	3.029 (5)	169
N8*B*—H8*B*···Cl2*B*	0.91	2.13	3.034 (5)	171
C6*B*—H6*B*···Cl2*B*	0.93	2.79	3.578 (8)	144
C10*A*—H10*A*···Cl1*A*^{i}	0.97	2.70	3.521 (7)	143
C10*B*—H11*B*···Cl2*B*^{ii}	0.97	2.73	3.516 (7)	138
C12*A*—H12*A*···Cl2*B*^{iii}	0.93	2.66	3.559 (8)	163
C12*B*—H12*B*···Cl1*A*^{iv}	0.93	2.66	3.565 (8)	165
C7*A*—H71*A*···Cl1*A*^{iv}	0.97	2.78	3.647 (7)	149
C7*B*—H72*B*···Cl2*B*^{iii}	0.97	2.82	3.693 (7)	150
C9*B*—H91*B*···Cl2*B*^{v}	0.96	2.80	3.639 (8)	146
C9*A*—H93*A*···Cl1*A*^{vi}	0.96	2.82	3.667 (8)	148

Symmetry codes: (i) $x-1, y, z$; (ii) $1+x, y, z$; (iii) $-x, \frac{1}{2}+y, z$; (iv) $1-x, y-\frac{1}{2}, z$; (v) $-1-x, \frac{1}{2}+y, z$; (vi) $2-x, y-\frac{1}{2}, z$.

Although all the H atoms could be located in a difference map, due to their less than perfect geometrical parameters, it was decided to place them in calculated positions and include them in the refinement in the riding-model approximation. One overall isotropic displacement parameter was refined for methyl-group H atoms and another for the rest of the H atoms. The final $U_{\rm iso}$ was 0.079 (9) Å^2 for the methyl H atoms and 0.089 (5) Å^2 for the remainder. The C—H distances range from 0.93 to 0.97 Å. The N—H distance is 0.91 Å. The value of the Flack (1983) parameter indicates that the crystal is an inversion twin; this is not surprising, given the occurrence of inversion pairs of molecules in this non-centrosymmetric space group

Data collection: *Picker Operating Manual* (Picker, 1967); cell refinement: *Picker Operating Manual*; data reduction: *DATRDN* (Stewart, 1976); program(s) used to solve structure: *SHELXS97*

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(Sheldrick, 1997; program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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