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Pargyline hydrochloride: a monoclinic form

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.016 Å
R factor = 0.072
wR factor = 0.206
Data-to-parameter ratio = 8.0

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

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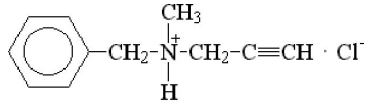
In the crystal structure of the title compound, *N*-benzyl-*N*-methylprop-2-yn-1-aminium chloride, $C_{11}H_{14}N^+\cdot Cl^-$, the asymmetric unit contains two independent enantiomeric cations related by a pseudo-center of symmetry. In both cations, the protonation occurs at the N atom, and the side chains are roughly perpendicular to the benzene rings. In addition to the conventional N—H···Cl hydrogen bonds, there are several weak hydrogen bonds of the type C—H···Cl. The cations are arranged head-to-head and tail-to-tail, producing hydrophilic and hydrophobic areas

Comment

As detailed in the preceding paper (Hempel *et al.*, 2005), pargyline is an irreversible inhibitor of monoamine oxidases. Crystallization of pargyline hydrochloride resulted in two crystal forms, each with two independent enantiomeric cations in the asymmetric unit.



(I)

The structure of the title compound, (I), is presented in Fig. 1. As in the orthorhombic structure, the asymmetric unit contains two independent enantiomeric molecules protonated at the N atom and related by a non-crystallographic inversion center, located in this crystal form at (0.83, 0.20, 0.64). The conformations of the two independent pargyline cations, as well as the two cations in the orthorhombic crystal form, are virtually identical; the torsion angles C1—C7—N8—C9 and C1—C7—N8—C10 differ by no more than 3.2 and 3.0°,

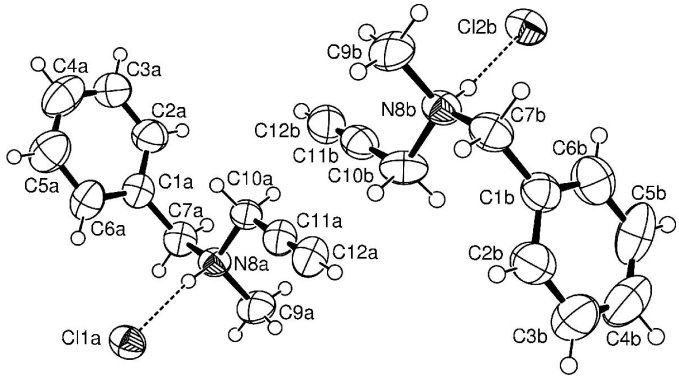


Figure 1
The structure of the asymmetric unit of pargyline hydrochloride, showing 50% probability displacement ellipsoids. H atoms are drawn as small circles of arbitrary radii. Hydrogen bonds are shown as dashed lines.

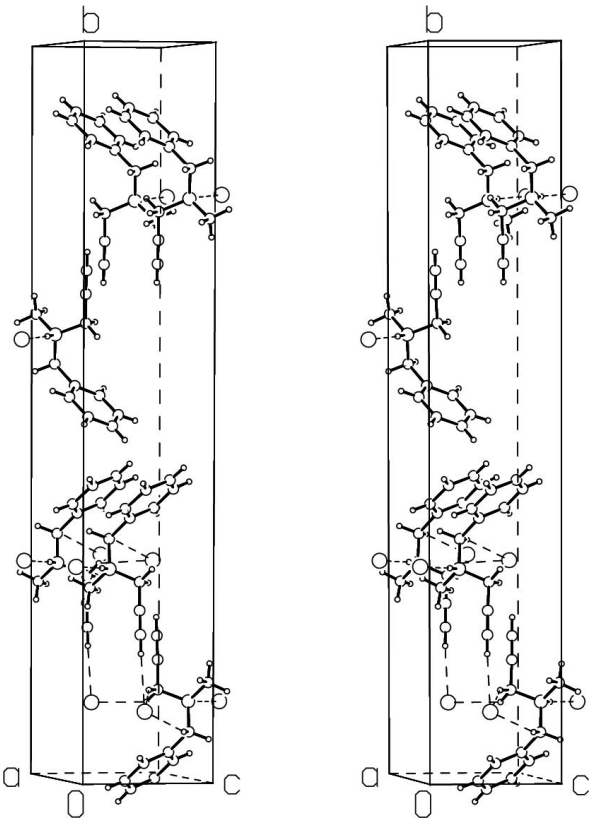


Figure 2
Stereoscopic view of the molecular packing and hydrogen-bond scheme (shown as dashed lines between atoms). Atoms are drawn as circles of arbitrary radii.

respectively, among the four cations. The production of the different crystal forms is therefore driven solely by alternative crystal-packing arrangements, in slightly different crystallization media. In both crystal structures, the cations are packed in a head-to-head and tail-to-tail fashion, creating distinct hydrophilic and hydrophobic regions, which run perpendicular to the *b* axis in this crystal form (Fig. 2).

Two standard N—H···Cl hydrogen bonds, and a variety of a weak non-standard hydrogen bonds of the type C—H···Cl (Steiner, 1997) define the coordination about the Cl^- ions. The difference here is that both H atoms at C10*B* are involved, *versus* only one in the orthorhombic form. The H atom at C6*B* does not participate here, and no C9 methyl groups H atoms are involved in this structure, whereas in the orthorhombic form, atom C6*B* and both methyl groups donate one H atom each to the hydrogen-bond network (Table 1). Van der Waals interactions also contribute to the crystal packing.

Experimental

Crystals of the monoclinic form of pargyline hydrochloride were obtained from a chloroform–benzene–butanone mixture (1:1:0.1) subjected to slow evaporation at 278 K. Crystals appeared after about two weeks. The crystals were small colorless needles of a rather poor quality. Crystallization trials to obtain better crystals were unsuccessful.

Crystal data

$C_{11}H_{14}N^+\cdot Cl^-$
M_r = 195.68
Monoclinic, $P2_1$
a = 5.739 (2) Å
b = 33.684 (5) Å
c = 6.003 (2) Å
β = 106.79 (4)°
V = 1111.0 (6) Å^3
Z = 4
D_x = 1.170 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 32 reflections
θ = 19–43°
μ = 2.67 mm^{-1}
T = 294 (2) K
Needle, colorless
0.28 × 0.09 × 0.05 mm

Data collection

Picker FACS-1 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.747, T_{max} = 0.870
2105 measured reflections
1918 independent reflections
1651 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 65.0°
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 39$
$l = 0 \rightarrow 7$
3 standard reflections every 100 reflections
intensity decay: 2.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.206
S = 1.52
1918 reflections
241 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1459P)^2 + 0.5682P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter = 0.41 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N8A—H8A···Cl1A	0.91	2.14	3.042 (7)	169
N8B—H8B···Cl2B	0.91	2.13	3.025 (6)	170
C10A—H10B···Cl1A^{i}	0.97	2.75	3.559 (8)	142
C10B—H10C···Cl2B^{ii}	0.97	2.75	3.531 (8)	138
C10B—H10D···Cl2B^{iii}	0.97	2.82	3.701 (8)	152
C12A—H12A···Cl2B^{ii}	0.93	2.64	3.546 (11)	164
C12B—H12B···Cl1A^{i}	0.93	2.66	3.559 (13)	163
C7A—H7A2···Cl1A^{iv}	0.97	2.80	3.648 (9)	147
C7B—H7B1···Cl2B^{iii}	0.97	2.82	3.669 (10)	147

Symmetry codes: (i) $x, y, 1+z$; (ii) $x, y, z-1$; (iii) $x-1, y, z-1$; (iv) $1+x, y, 1+z$.

Although all the H atoms could be located in a difference map, as in the case of the orthorhombic form, their poor behavior during the refinement forced us to place them in calculated positions and refine them 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_{iso} was 0.079 (10) for the methyl H atoms and 0.089 (7) for the remainder. The range of C—H distances is 0.93–0.97 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 and cell refinement: *Picker Operating Manual* (Picker, 1967); data reduction: *DATRDN* (Stewart, 1976); program(s) used to solve structure: *SHELXS97* (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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