

Nipecotic acid hydrochloride

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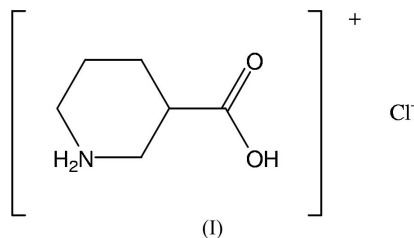
Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.022
 wR factor = 0.057
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound (3-carboxypiperidinium chloride), $\text{C}_6\text{H}_{12}\text{NO}_2^+\cdot\text{Cl}^-$, is the hydrochloride of nipecotic acid and is used as a drug intermediate and in the synthesis of γ -aminobutyric acid (GABA) uptake inhibitors. The geometric parameters are in the normal ranges. The crystal packing is stabilized by $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

Comment

Nipecotic acid or 3-piperidinecarboxylic acid is used as a drug intermediate and also in the synthesis of γ -aminobutyric acid (GABA) uptake inhibitors (Muralidhar *et al.*, 1994). A review on the neurochemical and behavioural profile of a derivative of nipecotic acid hydrochloride has been reported by Suzdak & Jansen (1995). In view of the importance of nipecotic acid, the present paper reports the crystal structure of nipecotic acid hydrochloride, (I).



A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; Mogul Version 1.0.1; Allen, 2002). The heterocycle adopts a chair conformation. The crystal packing is stabilized by $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

Experimental

Nipecotic acid was purchased from the Aldrich Chemical Company and was converted to its hydrochloride by adding a mixture of isopropyl alcohol and hydrochloric acid (80/20). The compound was recrystallized from ethanol.

Crystal data

 $\text{C}_6\text{H}_{12}\text{NO}_2^+\cdot\text{Cl}^-$
 $M_r = 165.62$
Monoclinic, $P2_1$
 $a = 7.2545$ (10) Å
 $b = 7.2018$ (9) Å
 $c = 7.7886$ (13) Å
 $\beta = 97.819$ (12)°
 $V = 403.14$ (10) Å³
 $Z = 2$ $D_x = 1.364$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 8363 reflections
 $\theta = 3.8$ – 25.7°
 $\mu = 0.42$ mm⁻¹
 $T = 173$ (2) K
Block, colourless
 $0.37 \times 0.23 \times 0.19$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)
 $T_{\min} = 0.861$, $T_{\max} = 0.925$
 3354 measured reflections

1477 independent reflections
 1460 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 25.5^\circ$
 $h = -8 \rightarrow 8$
 $k = -8 \rightarrow 8$
 $l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.057$
 $S = 1.10$
 1477 reflections
 104 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.0946P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.075 (10)
 Absolute structure: Flack (1983), 671 Friedel pairs
 Flack parameter: 0.10 (6)

Table 1

Selected geometric parameters (\AA , $^\circ$).

C2–N3	1.4972 (18)	C7–O2	1.187 (2)
N3–C4	1.502 (2)	C7–O1	1.323 (2)
C2–N3–C4	113.78 (12)	O2–C7–C1	123.39 (16)
O2–C7–O1	122.79 (15)	O1–C7–C1	113.68 (14)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1–H1O \cdots C11	0.89 (3)	2.11 (3)	2.9985 (13)	173 (2)
N3–H3A \cdots C11 ⁱ	0.96 (2)	2.20 (2)	3.1428 (15)	166 (2)
N3–H3B \cdots C11 ⁱⁱ	0.90 (2)	2.47 (2)	3.2525 (15)	147 (2)
N3–H3B \cdots O2 ⁱⁱ	0.90 (2)	2.42 (2)	2.9590 (19)	119 (2)

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

All H atoms were located in a difference map. Those bonded to C atoms were positioned geometrically and refined with fixed individual displacement parameters (set to 1.2 times U_{eq} of the parent C atom)

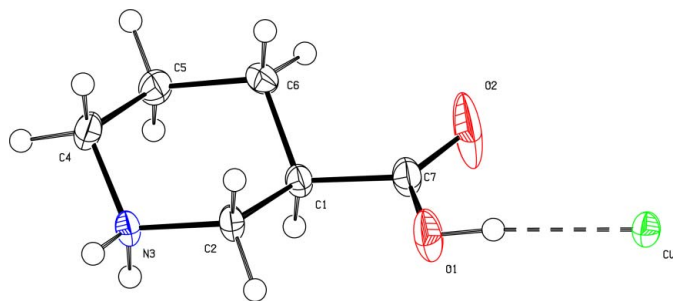


Figure 1

Perspective view of the title compound, showing the atom numbering and displacement ellipsoids drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

using a riding model, with C–H = 0.99 and 1.0 \AA for secondary and tertiary H atoms. The H atoms bonded to N and O atoms were refined freely. The ADDSYM routine in PLATON (Spek, 2003) detects a pseudo-centre of symmetry in the structure, which is fulfilled by approximately 80% of the structure. This would mean changing the space group from $P2_1$ to $P2_1/m$. In this case, the molecule must lie on a mirror plane. However, the molecule does not have any symmetry at all. Therefore, $P2_1$ is the correct space group and it is just a pseudo-centre of symmetry that PLATON detects.

Data collection: X-AREA (Stoe & Cie, 2001); cell refinement: X-AREA; data reduction: X-AREA; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PLATON.

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