

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Pyridine-4-carboximidamide chloride

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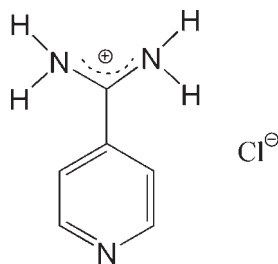
Received 27 July 2009; accepted 1 September 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.033; wR factor = 0.092; data-to-parameter ratio = 11.6.

In the title salt, $\text{C}_6\text{H}_8\text{N}_3^+\cdot\text{Cl}^-$, each pyridinecarboximidamide cation is linked to two symmetry-related cations through $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, and to two chloride ions by $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds. The $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds involve the pyridine N atom and one NH_2 group. In the crystal, $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds extend the structure into two-dimensional layers. Weak $\text{C}-\text{H}\cdots\text{Cl}$ interactions further connect these layers into a three-dimensional network.

Related literature

For background, see: Chudinov *et al.* (2005); Kamei *et al.* (2005).



Experimental

Crystal data

 $\text{C}_6\text{H}_8\text{N}_3^+\cdot\text{Cl}^-$ $M_r = 157.60$ Orthorhombic, $Pbca$ $a = 7.3928$ (13) Å $b = 10.4467$ (16) Å $c = 18.925$ (3) Å $V = 1461.6$ (4) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.44$ mm⁻¹ $T = 293$ K $0.37 \times 0.32 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

 $T_{\min} = 0.853$, $T_{\max} = 0.911$

1949 measured reflections

1435 independent reflections

1215 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.033$ $wR(F^2) = 0.092$ $S = 1.04$

1435 reflections

124 parameters

All H-atom parameters refined

 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{N1}^{\text{i}}$	0.88 (2)	2.22 (2)	3.058 (2)	160 (2)
$\text{N2}-\text{H2B}\cdots\text{Cl1}$	0.83 (2)	2.79 (2)	3.476 (2)	142 (2)
$\text{N3}-\text{H3A}\cdots\text{Cl1}$	0.93 (2)	2.19 (2)	3.100 (2)	167 (2)
$\text{N3}-\text{H3B}\cdots\text{Cl1}^{\text{ii}}$	0.89 (2)	2.41 (2)	3.270 (2)	161 (2)
$\text{C5}-\text{H5}\cdots\text{Cl1}^{\text{iii}}$	0.90 (2)	2.68 (2)	3.556 (2)	166 (2)

Symmetry codes: (i) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, z$; (iii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2242).

References

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- Kamei, K., Maeda, N., Katsuragi-Ogino, R., Koyama, M., Nakajima, M., Tatsuoka, T., Ohno, T. & Inoue, T. (2005). *Bioorg. Med. Chem. Lett.* **15**, 2990–2993.
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supplementary materials

Acta Cryst. (2009). E65, o2408 [doi:10.1107/S160053680903517X]

Pyridine-4-carboximidamide chloride

P. Fan, L. Wang and H. Zhang

Comment

The title compound, also known as isonicotinamide hydrochloride, served as a key intermediate in the synthesis of pharmacologically active compounds. It had attracted a great deal of interest during recent years. A series of new piperidinyl- and 1,2,3,6-tetrahydropyridinylpyrimidine derivatives was synthesized by using isonicotinamide as an important intermediate. Isonicotinamide has a unique structure and exists in the form of hydrochloride or acetate (Chudinov *et al.*, 2005; Kamei *et al.*, 2005).

The title compound is an organic salt (Fig. 1). In the cation, dihedral angle between the pyridyl ring and the plane confined by N2, N3 and C6 is 42.1°. Each isonicotinamide cation is connected to two other cations by N—H···N hydrogen bonds, and to two Cl⁻ anions by N—H···Cl hydrogen bonds (Fig. 2), to form two dimensional layers including one-dimensional zigzag chains (Fig. 3). Weak C—H···Cl interactions [C···Cl = 3.556 (2) Å] link these layers to provide a three-dimensional supramolecular network.

Experimental

The title compound was prepared according to the method of Kamei *et al.* (2005). Block-shaped crystals suitable for X-ray diffraction were obtained from ethanol/acetone.

Refinement

H atoms were located from difference maps and freely refined.

Figures

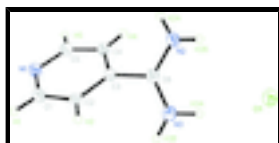


Fig. 1. View of (I), showing atomic labels and displacement ellipsoids drawn at 30% probability level.

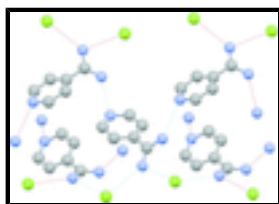


Fig. 2. N—H···N and N—H···Cl hydrogen bonds in the crystal.

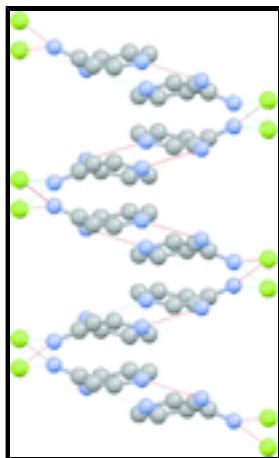


Fig. 3. View of the hydrogen bonded one-dimensional chain along *b* axis.

Pyridine-4-carboximidamide chloride

Crystal data

$C_6H_8N_3^+ \cdot Cl^-$

$M_r = 157.60$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 7.3928$ (13) Å

$b = 10.4467$ (16) Å

$c = 18.925$ (3) Å

$V = 1461.6$ (4) Å³

$Z = 8$

$F_{000} = 656$

$D_x = 1.432$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 542 reflections

$\theta = 2.3$ – 22.8°

$\mu = 0.44$ mm⁻¹

$T = 293$ K

Block, colourless

$0.37 \times 0.32 \times 0.21$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Monochromator: graphite

$T = 293$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2001)

$T_{\min} = 0.853$, $T_{\max} = 0.911$

1949 measured reflections

1435 independent reflections

1215 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 26.0^\circ$

$\theta_{\text{min}} = 2.2^\circ$

$h = -9 \rightarrow 1$

$k = -1 \rightarrow 12$

$l = -23 \rightarrow 1$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.5489P]$

$wR(F^2) = 0.092$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} = 0.001$
1435 reflections	$\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
124 parameters	$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0056 (15)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.0852 (2)	0.38353 (14)	0.75509 (8)	0.0367 (4)
C1	0.0499 (3)	0.36114 (17)	0.82329 (10)	0.0367 (4)
Cl1	0.15740 (8)	0.91636 (4)	1.05546 (2)	0.0432 (2)
N2	0.0353 (2)	0.79417 (15)	0.89141 (9)	0.0369 (4)
C2	0.0593 (3)	0.45387 (17)	0.87518 (9)	0.0341 (4)
N3	0.1766 (3)	0.66264 (17)	0.97084 (9)	0.0428 (4)
C3	0.1026 (2)	0.57807 (15)	0.85578 (9)	0.0285 (4)
C4	0.1401 (3)	0.60305 (17)	0.78520 (9)	0.0337 (4)
C5	0.1315 (3)	0.50297 (18)	0.73774 (9)	0.0376 (4)
C6	0.1052 (2)	0.68338 (16)	0.90895 (9)	0.0310 (4)
H4	0.171 (3)	0.6863 (18)	0.7679 (10)	0.033 (5)*
H1	0.015 (3)	0.275 (2)	0.8348 (11)	0.044 (6)*
H2	0.032 (3)	0.4327 (18)	0.9204 (11)	0.041 (5)*
H5	0.159 (3)	0.519 (2)	0.6925 (12)	0.048 (6)*
H2B	0.037 (3)	0.854 (2)	0.9202 (13)	0.060 (7)*
H2A	-0.016 (3)	0.803 (2)	0.8500 (12)	0.050 (6)*
H3B	0.229 (4)	0.590 (2)	0.9833 (12)	0.056 (7)*
H3A	0.184 (3)	0.732 (2)	1.0012 (14)	0.061 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0452 (9)	0.0312 (8)	0.0338 (8)	0.0019 (7)	0.0001 (7)	-0.0051 (7)
C1	0.0439 (11)	0.0261 (9)	0.0400 (10)	-0.0002 (8)	-0.0007 (9)	0.0007 (7)
Cl1	0.0580 (3)	0.0357 (3)	0.0359 (3)	0.0007 (2)	-0.0055 (2)	-0.00566 (18)
N2	0.0484 (10)	0.0262 (8)	0.0360 (9)	0.0009 (7)	0.0006 (8)	-0.0043 (7)
C2	0.0436 (11)	0.0306 (9)	0.0280 (9)	0.0035 (8)	0.0027 (8)	0.0019 (7)
N3	0.0613 (12)	0.0348 (9)	0.0323 (8)	0.0070 (8)	-0.0090 (8)	-0.0067 (7)
C3	0.0313 (9)	0.0266 (8)	0.0277 (8)	0.0021 (7)	-0.0020 (7)	-0.0021 (7)
C4	0.0421 (10)	0.0280 (9)	0.0310 (9)	-0.0020 (8)	0.0003 (8)	0.0024 (7)
C5	0.0488 (12)	0.0377 (10)	0.0264 (9)	-0.0002 (8)	0.0022 (8)	-0.0012 (8)
C6	0.0348 (9)	0.0280 (9)	0.0300 (9)	-0.0018 (7)	0.0031 (7)	-0.0020 (7)

supplementary materials

Geometric parameters (\AA , $^\circ$)

N1—C5	1.335 (2)	N3—C6	1.303 (2)
N1—C1	1.337 (2)	N3—H3B	0.89 (2)
C1—C2	1.381 (3)	N3—H3A	0.93 (3)
C1—H1	0.96 (2)	C3—C4	1.389 (2)
N2—C6	1.310 (2)	C3—C6	1.491 (2)
N2—H2B	0.83 (3)	C4—C5	1.380 (3)
N2—H2A	0.88 (2)	C4—H4	0.958 (19)
C2—C3	1.386 (2)	C5—H5	0.90 (2)
C2—H2	0.91 (2)		
C5—N1—C1	116.80 (15)	C2—C3—C4	118.47 (16)
N1—C1—C2	123.63 (17)	C2—C3—C6	120.99 (15)
N1—C1—H1	115.7 (12)	C4—C3—C6	120.52 (15)
C2—C1—H1	120.6 (12)	C5—C4—C3	118.33 (17)
C6—N2—H2B	119.8 (17)	C5—C4—H4	118.5 (11)
C6—N2—H2A	119.2 (15)	C3—C4—H4	123.2 (11)
H2B—N2—H2A	121 (2)	N1—C5—C4	124.05 (17)
C1—C2—C3	118.68 (16)	N1—C5—H5	118.2 (15)
C1—C2—H2	119.3 (13)	C4—C5—H5	117.7 (15)
C3—C2—H2	122.0 (13)	N3—C6—N2	122.31 (17)
C6—N3—H3B	123.9 (15)	N3—C6—C3	119.28 (16)
C6—N3—H3A	116.9 (16)	N2—C6—C3	118.41 (16)
H3B—N3—H3A	119 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots N1 ⁱ	0.88 (2)	2.22 (2)	3.058 (2)	160 (2)
N3—H3A \cdots C11	0.93 (2)	2.19 (2)	3.100 (2)	167 (2)
N2—H2B \cdots C11	0.83 (2)	2.79 (2)	3.476 (2)	142 (2)
N3—H3B \cdots C11 ⁱⁱ	0.89 (2)	2.41 (2)	3.270 (2)	161 (2)
C5—H5 \cdots C11 ⁱⁱⁱ	0.90 (2)	2.68 (2)	3.556 (2)	166 (2)

Symmetry codes: (i) $-x, y+1/2, -z+3/2$; (ii) $-x+1/2, y-1/2, z$; (iii) $x, -y+3/2, z-1/2$.

Fig. 1

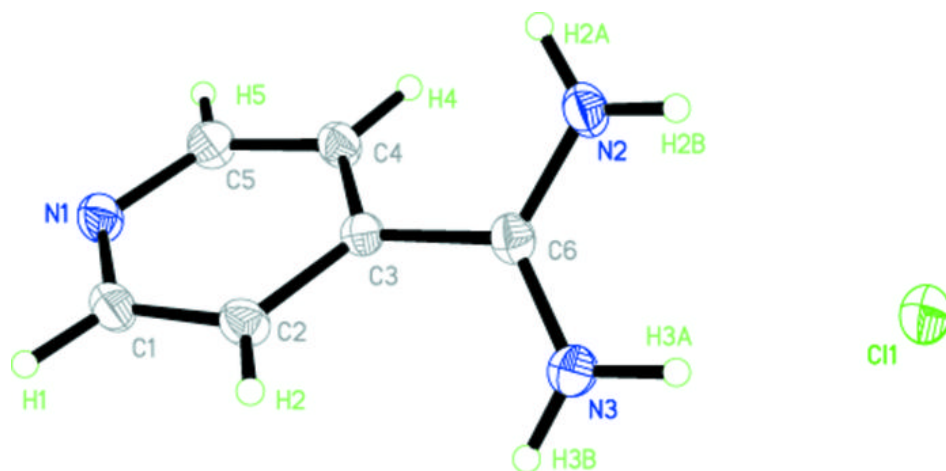


Fig. 2

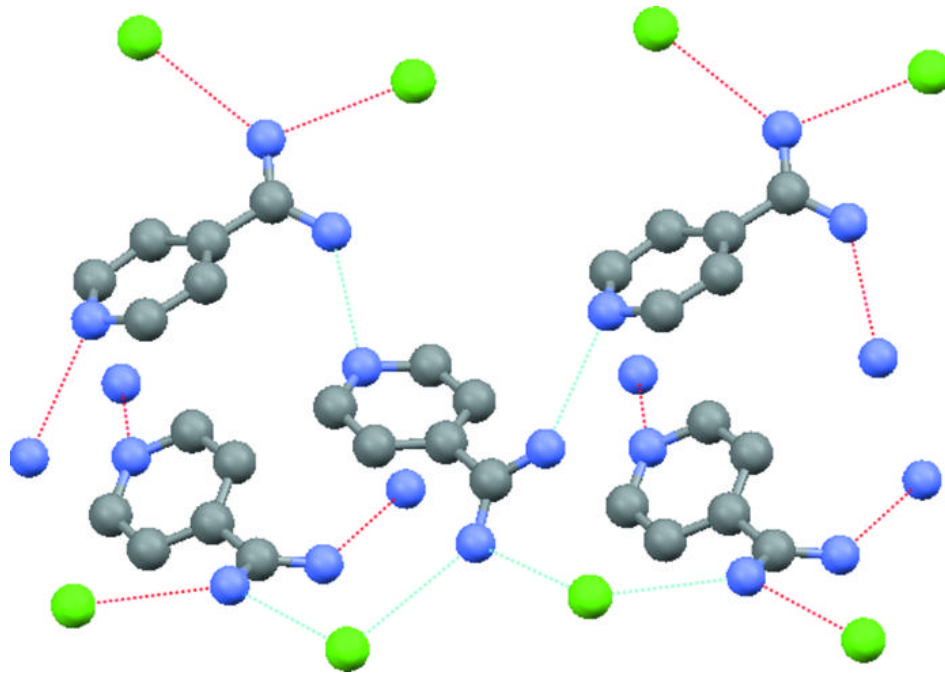


Fig. 3

