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Pyridine-4-carboximidamidate chloride

Ping Fan,[a]* Lei Wang[a] and Huidong Zhang[b]

[a]College of Chemistry, Liaoning University, Shenyang 110036, People's Republic of China, and [b]Liaoning Fixed Star Chemicals (Group) Co. Ltd, Dandong 118000, People's Republic of China
Correspondence e-mail: pingfan@lnu.edu.cn

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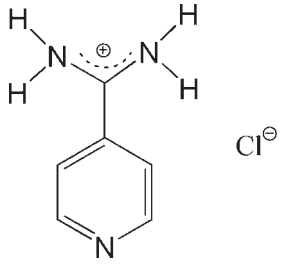
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.092; data-to-parameter ratio = 11.6.

In the title salt, $C_6H_8N_3^+\cdot Cl^-$, each pyridinecarboximidamidate cation is linked to two symmetry-related cations through N—H···N hydrogen bonds, and to two chloride ions by N—H···Cl hydrogen bonds. The N—H···N hydrogen bonds involve the pyridine N atom and one NH_2 group. In the crystal, N—H···N and N—H···Cl hydrogen bonds extend the structure into two-dimensional layers. Weak C—H···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

$C_6H_8N_3^+\cdot Cl^-$ 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) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 293 K
0.37 × 0.32 × 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_{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
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1^{i}	0.88 (2)	2.22 (2)	3.058 (2)	160 (2)
N2—H2B···Cl1	0.83 (2)	2.79 (2)	3.476 (2)	142 (2)
N3—H3A···Cl1	0.93 (2)	2.19 (2)	3.100 (2)	167 (2)
N3—H3B···Cl1^{ii}	0.89 (2)	2.41 (2)	3.270 (2)	161 (2)
C5—H5···Cl1^{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

Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chudinov, M. V., Konstantinova, I. D., Ryzhova, O. I., Esipov, R. S., Yurkevich, A. M., Shvets, V. I. & Miroshnikov, A. I. (2005). *Pharm. Chem. J.* **39**, 212–215.
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.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2408 [doi:10.1107/S160053680903517X]

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S1. Comment

The title compound, also known as isonicotinamidine 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 isonicotinamidine as an important intermediate. Isonicotinamidine 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 isonicotinamidine 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.

S2. 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.

S3. Refinement

H atoms were located from difference maps and freely refined.

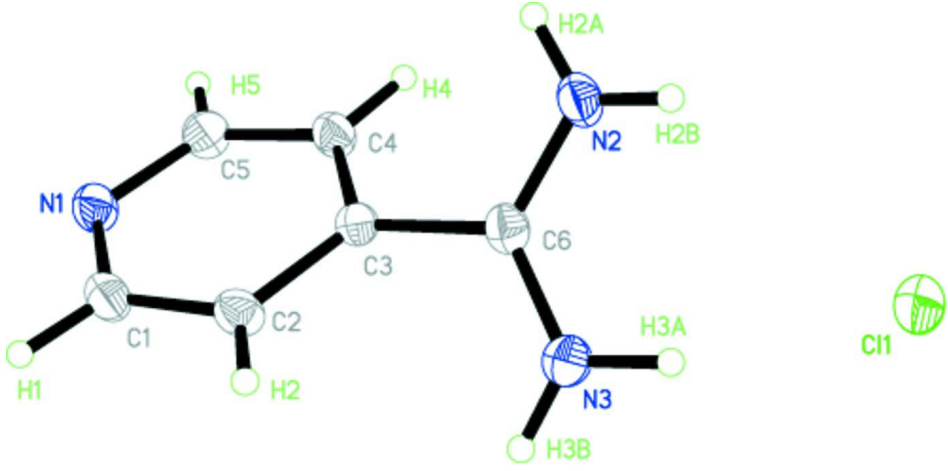

Figure 1

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

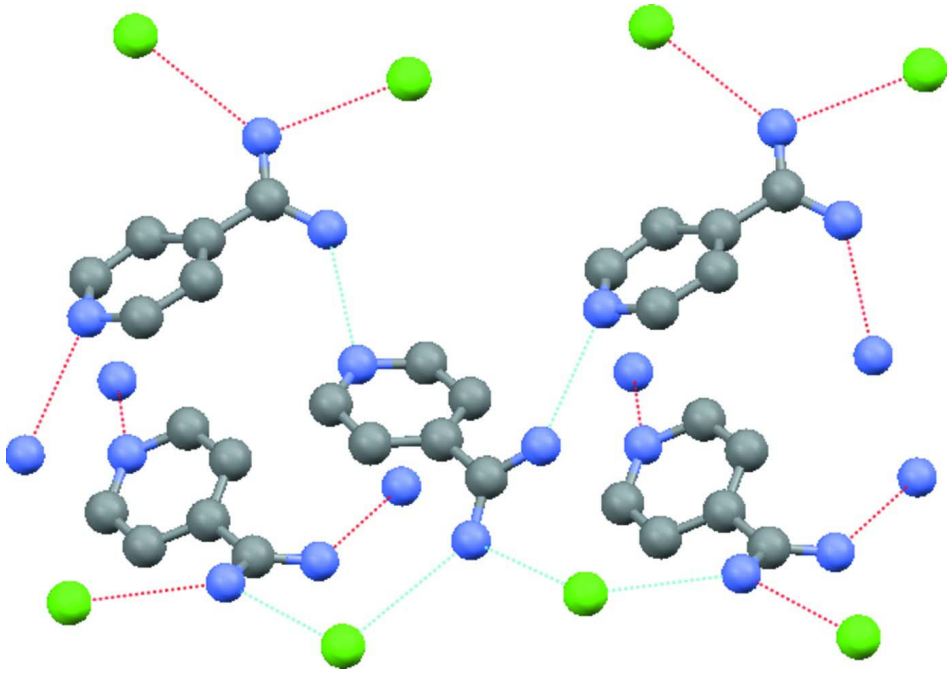

Figure 2

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

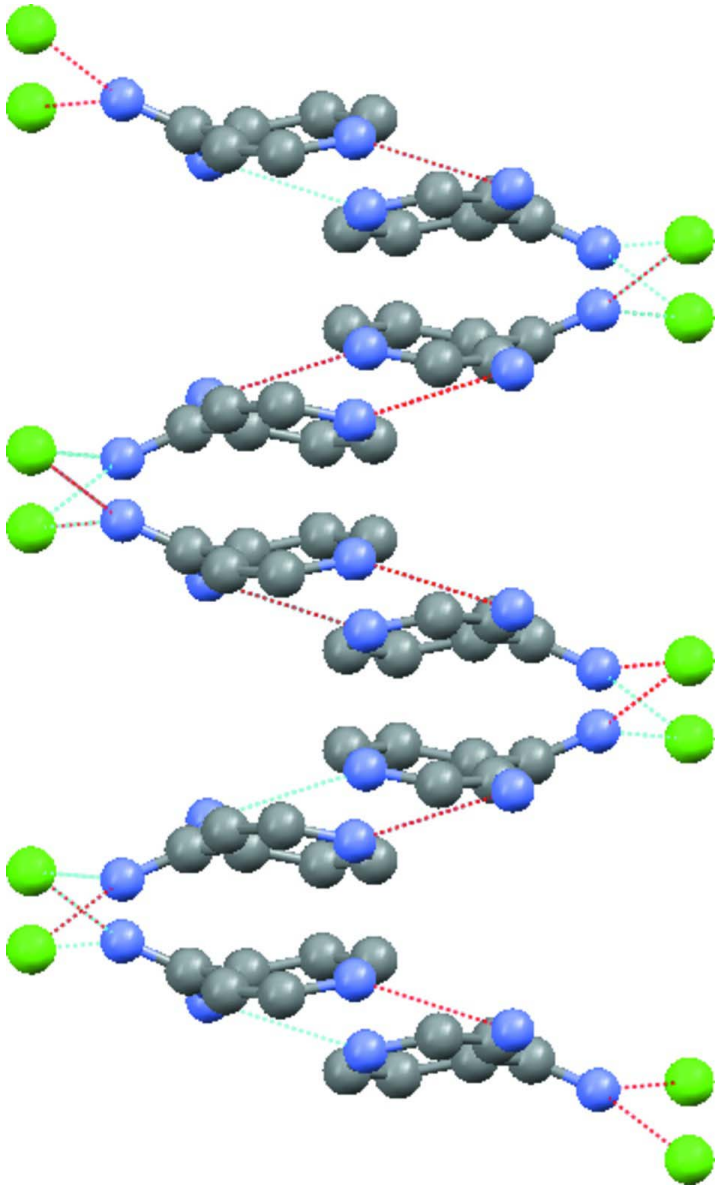

Figure 3

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

Pyridine-4-carboximidamidate 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) Å^3
Z = 8
F(000) = 656
D_x = 1.432 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 542 reflections
θ = 2.3–22.8°
μ = 0.44 mm^{-1}
T = 293 K
Block, colourless
0.37 × 0.32 × 0.21 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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_{int} = 0.018
θ_{max} = 26.0°, θ_{min} = 2.2°
$h = -9\rightarrow1$
$k = -1\rightarrow12$
$l = -23\rightarrow1$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.04
1435 reflections
124 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.5489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0056 (15)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)

Geometric parameters (Å, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N1^{i}	0.88 (2)	2.22 (2)	3.058 (2)	160 (2)
N3—H3*A*···Cl1	0.93 (2)	2.19 (2)	3.100 (2)	167 (2)
N2—H2*B*···Cl1	0.83 (2)	2.79 (2)	3.476 (2)	142 (2)
N3—H3*B*···Cl1^{ii}	0.89 (2)	2.41 (2)	3.270 (2)	161 (2)
C5—H5···Cl1^{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.