

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-Cyanopyridinium chloride

Wen-Ni Zheng

College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China

Correspondence e-mail: chenxinyuanseu@yahoo.com.cn

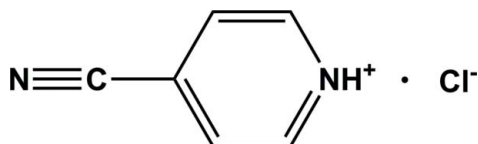
Received 25 April 2012; accepted 25 April 2012

 Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.086; wR factor = 0.234; data-to-parameter ratio = 15.0.

In the crystal structure of the title salt, $\text{C}_6\text{H}_5\text{N}_2^+\cdot\text{Cl}^-$, the pyridinium cation links to the Cl^- anion via an $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond. Weak $\text{C}-\text{H}\cdots\text{Cl}$ interactions also occur.

Related literature

For the structures and properties of related compounds, see: Chen *et al.* (2000); Dai & Chen (2011); Xu *et al.* (2011); Liu *et al.* (1999); Zhao *et al.* (2003); Zheng (2011).



Experimental

Crystal data

 $\text{C}_6\text{H}_5\text{N}_2^+\cdot\text{Cl}^-$
 $M_r = 140.57$

 Triclinic, $P\bar{1}$
 $a = 6.6166$ (2) Å

 $b = 7.6552$ (3) Å

 $c = 8.3495$ (5) Å

 $\alpha = 63.957$ (5)°

 $\beta = 69.830$ (2)°

 $\gamma = 74.367$ (4)°

 $V = 353.16$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 0.45$ mm⁻¹
 $T = 123$ K

 $0.10 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 diffractometer

Absorption correction: multi-scan

 (*CrystalClear*; Rigaku, 2005)

 $T_{\min} = 0.910$, $T_{\max} = 1.000$

3077 measured reflections

1231 independent reflections

 1078 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.086$
 $wR(F^2) = 0.234$
 $S = 1.32$

1231 reflections

82 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.97$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.62$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{Cl1}^{\text{i}}$	0.90	2.14	3.033 (5)	174
$\text{C4}-\text{H4A}\cdots\text{Cl1}$	0.95	2.71	3.566 (5)	151
$\text{C5}-\text{H5A}\cdots\text{Cl1}^{\text{ii}}$	0.95	2.65	3.566 (6)	161

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by a start-up grant from Southeast University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5524).

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supporting information

Acta Cryst. (2012). E68, o1611 [doi:10.1107/S1600536812018648]

4-Cyanopyridinium chloride

Wen-Ni Zheng

S1. Comment

Simple organic salts containing strong intramolecular H-bonds have attracted an attention as materials which display ferroelectric-paraelectric phase transitions (Chen *et al.*, 2000; Liu *et al.*, 1999; Zhao *et al.*, 2003). With the purpose of obtaining phase transition crystals of organic salts, various organic molecules have been studied and a series of new materials have been elaborated (Dai & Chen, 2011; Xu *et al.*, 2011; Zheng, 2011). Herewith we present the synthesis and crystal structure of the title compound.

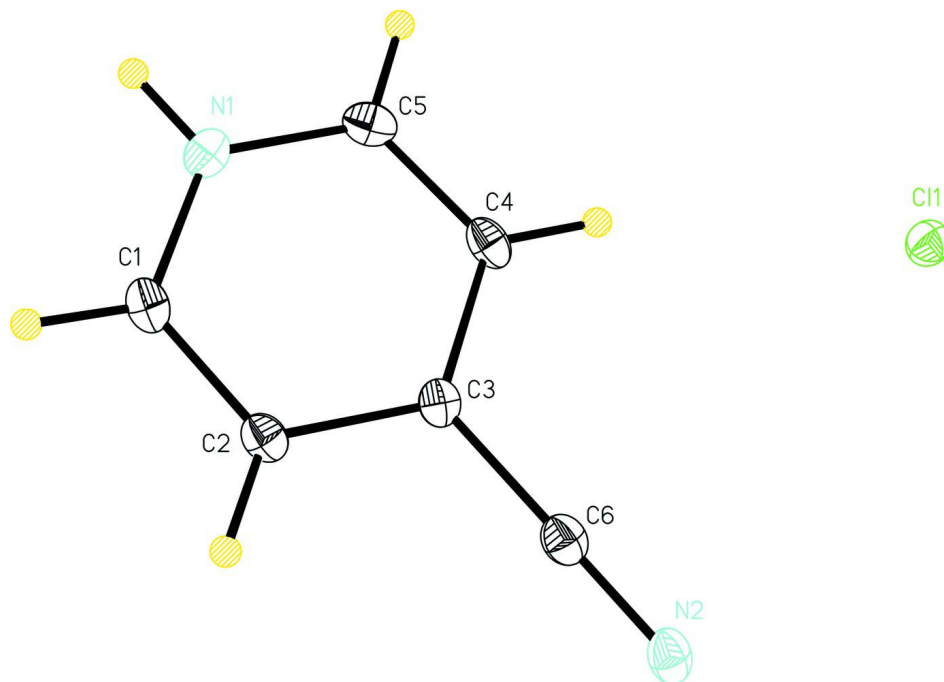
In the title compound (Fig. 1), the bond lengths and angles have normal values. The asymmetric unit was composed of one 4-cyanopyridinium cation and one Cl⁻ anion. The protonated N atom was involved in strong intramolecular N—H \cdots Cl hydrogen bonds with the N \cdots Cl distance of 3.033 (5) Å. The weak intermolecular C4—H4A \cdots Cl1 and C5—H45 \cdots Cl1 interactions were presented in the crystal structure with C5 \cdots Cl1 = 3.566 (5) Å and C5 \cdots Cl1 = 3.566 (6) Å, respectively. The crystal packing is further stabilized by aromatic $\pi\cdots\pi$ interactions between the pyridine rings of the neighbouring 4-cyanopyridinium cations with the Cg \cdots Cg distances of 4.416 (5) Å and 4.102 (5) Å (Cg is the centroid of the pyridine ring) (Fig. 2 and Table 1).

S2. Experimental

The HCl (5 mL), isonicotinonitrile (20 mmol) and ethanol (50 mL) were added into a 100 mL flask. The mixture was stirred at 333 K for 2 h, and then the precipitate was filtrated out. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution.

S3. Refinement

All the H atoms were situated into the idealized positions and treated as riding with C—H = 0.95 and N—H = 0.90 Å, $U_{iso}(\text{H}) = 1.2U_{eq}(\text{C,N})$.

**Figure 1**

A view of the asymmetric unit with the atomic numbering scheme. The displacement ellipsoids were drawn at the 30% probability level.

4-Cyanopyridinium chloride

Crystal data

$C_6H_5N_2^+ \cdot Cl^-$

$M_r = 140.57$

Triclinic, $P1$

Hall symbol: $-P 1$

$a = 6.6166$ (2) Å

$b = 7.6552$ (3) Å

$c = 8.3495$ (5) Å

$\alpha = 63.957$ (5)°

$\beta = 69.830$ (2)°

$\gamma = 74.367$ (4)°

$V = 353.16$ (3) Å³

$Z = 2$

$F(000) = 144$

$D_x = 1.322$ Mg m⁻³

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

Cell parameters from 1231 reflections

$\theta = 2.8$ – 27.5 °

$\mu = 0.45$ mm⁻¹

$T = 123$ K

Block, colorless

$0.10 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels mm⁻¹

CCD profile fitting scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.910$, $T_{\max} = 1.000$

3077 measured reflections

1231 independent reflections

1078 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 2.8$ °

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.086$
 $wR(F^2) = 0.234$
 $S = 1.32$
 1231 reflections
 82 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0002P)^2 + 3.2997P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.97 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.62 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.8017 (7)	0.1582 (6)	0.6278 (6)	0.0267 (11)
N1	0.4202 (7)	0.2736 (6)	0.0960 (6)	0.0234 (10)
H1	0.3576	0.2900	0.0094	0.028*
C1	0.6318 (8)	0.1817 (7)	0.0856 (7)	0.0238 (12)
H1A	0.7092	0.1377	-0.0122	0.029*
C3	0.6118 (8)	0.2221 (7)	0.3646 (6)	0.0192 (11)
C4	0.3919 (8)	0.3182 (7)	0.3713 (7)	0.0238 (13)
H4A	0.3103	0.3648	0.4667	0.029*
C6	0.7156 (8)	0.1908 (7)	0.5071 (7)	0.0258 (13)
C5	0.3016 (8)	0.3411 (7)	0.2334 (7)	0.0251 (13)
H5A	0.1565	0.4042	0.2349	0.030*
C2	0.7328 (8)	0.1531 (7)	0.2198 (7)	0.0222 (12)
H2A	0.8783	0.0894	0.2146	0.027*
Cl1	0.1808 (2)	0.31778 (18)	0.82634 (17)	0.0249 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.031 (2)	0.021 (2)	0.0189 (19)	-0.0006 (18)	-0.0026 (17)	-0.0048 (16)
N1	0.031 (2)	0.0187 (18)	0.0204 (18)	-0.0053 (17)	-0.0098 (16)	-0.0034 (15)
C1	0.028 (2)	0.017 (2)	0.020 (2)	-0.001 (2)	-0.0027 (19)	-0.0056 (18)
C3	0.024 (2)	0.0126 (19)	0.015 (2)	-0.0054 (18)	-0.0029 (18)	-0.0008 (16)
C4	0.023 (2)	0.017 (2)	0.022 (2)	0.0001 (19)	0.0000 (19)	-0.0059 (18)
C6	0.025 (2)	0.023 (2)	0.025 (2)	-0.002 (2)	-0.003 (2)	-0.0097 (19)
C5	0.022 (2)	0.018 (2)	0.029 (2)	-0.0011 (19)	-0.008 (2)	-0.0029 (19)

C2	0.023 (2)	0.016 (2)	0.022 (2)	0.0010 (19)	-0.0046 (18)	-0.0052 (18)
Cl1	0.0244 (6)	0.0273 (6)	0.0235 (5)	0.0001 (5)	-0.0081 (4)	-0.0108 (4)

Geometric parameters (Å, °)

N2—C6	1.225 (7)	C3—C4	1.437 (7)
N1—C5	1.371 (7)	C3—C6	1.473 (8)
N1—C1	1.379 (6)	C4—C5	1.398 (8)
N1—H1	0.8999	C4—H4A	0.9500
C1—C2	1.404 (8)	C5—H5A	0.9500
C1—H1A	0.9500	C2—H2A	0.9500
C3—C2	1.430 (7)		
C5—N1—C1	122.5 (5)	C5—C4—H4A	121.0
C5—N1—H1	118.8	C3—C4—H4A	121.0
C1—N1—H1	118.8	N2—C6—C3	177.9 (5)
N1—C1—C2	119.9 (5)	N1—C5—C4	120.7 (4)
N1—C1—H1A	120.0	N1—C5—H5A	119.6
C2—C1—H1A	120.0	C4—C5—H5A	119.6
C2—C3—C4	120.3 (5)	C1—C2—C3	118.5 (4)
C2—C3—C6	118.9 (4)	C1—C2—H2A	120.7
C4—C3—C6	120.8 (5)	C3—C2—H2A	120.7
C5—C4—C3	118.1 (5)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots Cl1 ⁱ	0.90	2.14	3.033 (5)	174
C4—H4A \cdots Cl1	0.95	2.71	3.566 (5)	151
C5—H5A \cdots Cl1 ⁱⁱ	0.95	2.65	3.566 (6)	161

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