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4-Amino-3-ammoniopyridinium dichloride

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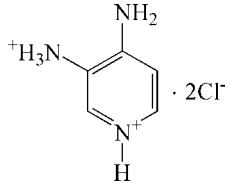
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.068; data-to-parameter ratio = 16.2.

The anions and cations of the title compound, $C_5H_9N_3^{2+}\cdot 2Cl^-$, are connected by two chloride-bridged three-centered N—H···Cl hydrogen bonds into a three-dimensional network. The aromatic rings are not involved in stacking interactions.

Related literature

For bond distances and angles in pyridine, derived from microwave spectra, see: Sørensen *et al.* (1974). For details of the N—H···Cl hydrogen bond in 4,4′-bipyridine compounds, see: Iyere *et al.* (2003). For N—H···Cl and secondary interactions in pyridinium chlorides, see: Jones *et al.* (2002); in 4-acetylpyridinium chloride, see: Kochel (2005). For N—H···Cl and O—H···Cl contacts in a triphenyl-pyridinium chloride (1/1) adduct, see: Sykora & Cioffi (2007).



Experimental

Crystal data

$C_5H_9N_3^{2+}\cdot 2Cl^-$
M_r = 182.05
Monoclinic, $P2_1/c$
a = 8.362 (2) Å
b = 7.3218 (19) Å
c = 13.239 (3) Å
β = 92.065 (4)°
V = 810.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 296 (2) K
0.41 × 0.31 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.734, T_{max} = 0.948
3949 measured reflections
1494 independent reflections
1345 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.068
S = 1.14
1494 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl2^{i}	0.89	2.22	3.1142 (15)	178
N1—H1B···Cl2^{ii}	0.89	2.37	3.1754 (16)	151
N1—H1C···Cl1^{iii}	0.89	2.23	3.0790 (16)	160
N2—H2A···Cl1^{ii}	0.86	2.39	3.2188 (17)	163
N2—H2B···Cl1^{iv}	0.86	2.42	3.2672 (17)	168
N3—H3···Cl2	0.86	2.59	3.2499 (16)	135
N3—H3···Cl2^{v}	0.86	2.70	3.3198 (16)	130

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2142).

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supplementary materials

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4-Amino-3-ammoniopyridinium dichloride

J.-H. Qin and J.-G. Wang

Comment

The title compound is a salt containing a diprotonated 3,4-diaminopyridine cation and two Cl^- anions (Fig. 1). The C1—N3—C5 bond angle is wider than that in pyridine (116.94 (3)°; Sørensen *et al.*, 1974) which indicates that the pyridine ring N atom is protonated (Table 1). Also, the 4-amino N atom is protonated. The projection of the crystal packing along the *b* axis is shown in Fig. 2. The Cl^- anions and the 3,4-diaminopyridinium cations in the title compound are bonded by two chlorine-bridged, three-centered N—H···Cl hydrogen bonds into a three-dimensional network (Fig. 2, Table 2). Example structures of related compounds with two- and three-centered N—H···Cl hydrogen bonds are discussed by Iyere *et al.* (2003); Jones *et al.* (2002); Kochel (2005) and Sykora & Cioffi (2007).

Experimental

3,4-diaminopyridine (0.01 mmol) and HCl (0.02 mmol) in 10 ml ethanol. Suitable crystals for X-ray analysis, were grown by allowing the solution to slowly evaporate for 15 days, and were subsequently filtered off, washed with methanol and dried under air.

Refinement

H atoms were constrained to idealized positions and refined using a riding model, with C—H distances of 0.93 Å [U_{iso}(H) = 1.2U_{eq}(C)], and NH distances of 0.86 Å for NH2 [U_{iso}(H) = 1.2U_{eq}(N)] and 0.89 Å for NH3 [U_{iso}(H) = 1.5U_{eq}(N)].

Figures

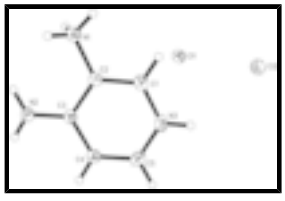

Fig. 1. A view of the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. A view of the title compound packing down the *b* axis.

4-Amino-3-ammoniopyridinium dichloride

Crystal data

$C_5H_9N_3^{2+}\cdot 2Cl^-$ F_{000} = 376

M_r = 182.05	D_x = 1.493 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2439 reflections
a = 8.362 (2) Å	θ = 3.1–28.2°
b = 7.3218 (19) Å	μ = 0.73 mm^{-1}
c = 13.239 (3) Å	T = 296 (2) K
β = 92.065 (4)°	Block, colorless
V = 810.0 (4) Å^3	0.41 × 0.31 × 0.07 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1494 independent reflections
Radiation source: fine-focus sealed tube	1345 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.014
T = 296(2) K	θ_{max} = 25.5°
φ and ω scans	θ_{min} = 2.4°
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −10→9
T_{min} = 0.734, T_{max} = 0.948	k = −6→8
3949 measured reflections	l = −16→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.025	H-atom parameters constrained
$wR(F^2)$ = 0.068	$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 0.2927P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.001
1494 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
92 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.05806 (5)	0.10977 (6)	0.30663 (3)	0.03884 (15)
Cl2	0.51526 (5)	0.00029 (6)	0.35359 (3)	0.03603 (14)
N1	0.26647 (16)	0.68257 (19)	0.30328 (10)	0.0320 (3)
H1A	0.3291	0.6281	0.2594	0.048*
H1B	0.3026	0.7949	0.3160	0.048*
H1C	0.1670	0.6887	0.2772	0.048*

N2	0.11914 (19)	0.8105 (2)	0.48188 (12)	0.0439 (4)
H2A	0.1182	0.8773	0.4284	0.053*
H2B	0.0737	0.8485	0.5352	0.053*
N3	0.33856 (18)	0.3102 (2)	0.48536 (11)	0.0376 (4)
H3	0.3861	0.2061	0.4869	0.045*
C1	0.3383 (2)	0.4103 (2)	0.40010 (13)	0.0329 (4)
H1	0.3866	0.3649	0.3431	0.039*
C2	0.26762 (18)	0.5778 (2)	0.39701 (12)	0.0271 (3)
C3	0.19067 (19)	0.6488 (2)	0.48205 (12)	0.0296 (4)
C4	0.1921 (2)	0.5357 (2)	0.56891 (13)	0.0366 (4)
H4	0.1422	0.5751	0.6267	0.044*
C5	0.2655 (2)	0.3703 (2)	0.56862 (14)	0.0391 (4)
H5	0.2656	0.2976	0.6262	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0414 (3)	0.0428 (3)	0.0324 (2)	0.00951 (19)	0.00267 (18)	−0.00166 (18)
Cl2	0.0410 (3)	0.0298 (2)	0.0379 (2)	0.00388 (17)	0.01080 (18)	0.00231 (17)
N1	0.0335 (7)	0.0344 (8)	0.0283 (7)	0.0006 (6)	0.0044 (6)	−0.0002 (6)
N2	0.0617 (10)	0.0360 (8)	0.0348 (8)	0.0202 (8)	0.0147 (7)	0.0032 (7)
N3	0.0403 (8)	0.0265 (7)	0.0462 (9)	0.0089 (6)	0.0023 (7)	0.0007 (6)
C1	0.0319 (9)	0.0323 (9)	0.0346 (9)	0.0018 (7)	0.0031 (7)	−0.0054 (7)
C2	0.0260 (8)	0.0283 (8)	0.0270 (8)	−0.0013 (6)	0.0012 (6)	−0.0008 (6)
C3	0.0309 (8)	0.0274 (8)	0.0306 (8)	0.0029 (7)	0.0017 (7)	−0.0016 (7)
C4	0.0434 (10)	0.0377 (10)	0.0291 (9)	0.0069 (8)	0.0069 (7)	0.0021 (7)
C5	0.0459 (10)	0.0366 (10)	0.0349 (10)	0.0038 (8)	0.0013 (8)	0.0077 (8)

Geometric parameters (Å, °)

N1—C2	1.458 (2)	N3—H3	0.8600
N1—H1A	0.8900	C1—C2	1.361 (2)
N1—H1B	0.8900	C1—H1	0.9300
N1—H1C	0.8900	C2—C3	1.416 (2)
N2—C3	1.326 (2)	C3—C4	1.417 (2)
N2—H2A	0.8600	C4—C5	1.358 (3)
N2—H2B	0.8600	C4—H4	0.9300
N3—C1	1.346 (2)	C5—H5	0.9300
N3—C5	1.353 (2)		
C2—N1—H1A	109.5	C2—C1—H1	119.9
C2—N1—H1B	109.5	C1—C2—C3	121.06 (15)
H1A—N1—H1B	109.5	C1—C2—N1	119.26 (14)
C2—N1—H1C	109.5	C3—C2—N1	119.65 (14)
H1A—N1—H1C	109.5	N2—C3—C2	122.95 (15)
H1B—N1—H1C	109.5	N2—C3—C4	120.92 (15)
C3—N2—H2A	120.0	C2—C3—C4	116.12 (15)
C3—N2—H2B	120.0	C5—C4—C3	120.64 (16)
H2A—N2—H2B	120.0	C5—C4—H4	119.7

C1—N3—C5	121.29 (15)	C3—C4—H4	119.7
C1—N3—H3	119.4	N3—C5—C4	120.64 (16)
C5—N3—H3	119.4	N3—C5—H5	119.7
N3—C1—C2	120.23 (16)	C4—C5—H5	119.7
N3—C1—H1	119.9		
C5—N3—C1—C2	1.9 (3)	N1—C2—C3—C4	177.72 (15)
N3—C1—C2—C3	−1.2 (2)	N2—C3—C4—C5	179.99 (18)
N3—C1—C2—N1	−178.99 (14)	C2—C3—C4—C5	0.7 (3)
C1—C2—C3—N2	−179.35 (16)	C1—N3—C5—C4	−1.2 (3)
N1—C2—C3—N2	−1.6 (2)	C3—C4—C5—N3	−0.1 (3)
C1—C2—C3—C4	−0.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl2^{i}	0.89	2.22	3.1142 (15)	178
N1—H1B···Cl2^{ii}	0.89	2.37	3.1754 (16)	151
N1—H1C···Cl1^{iii}	0.89	2.23	3.0790 (16)	160
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N2—H2B···Cl1^{iv}	0.86	2.42	3.2672 (17)	168
N3—H3···Cl2	0.86	2.59	3.2499 (16)	135
N3—H3···Cl2^{v}	0.86	2.70	3.3198 (16)	130

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, *y*+1, *z*; (iii) −*x*, *y*+1/2, −*z*+1/2; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*+1, −*y*, −*z*+1.

Fig. 1

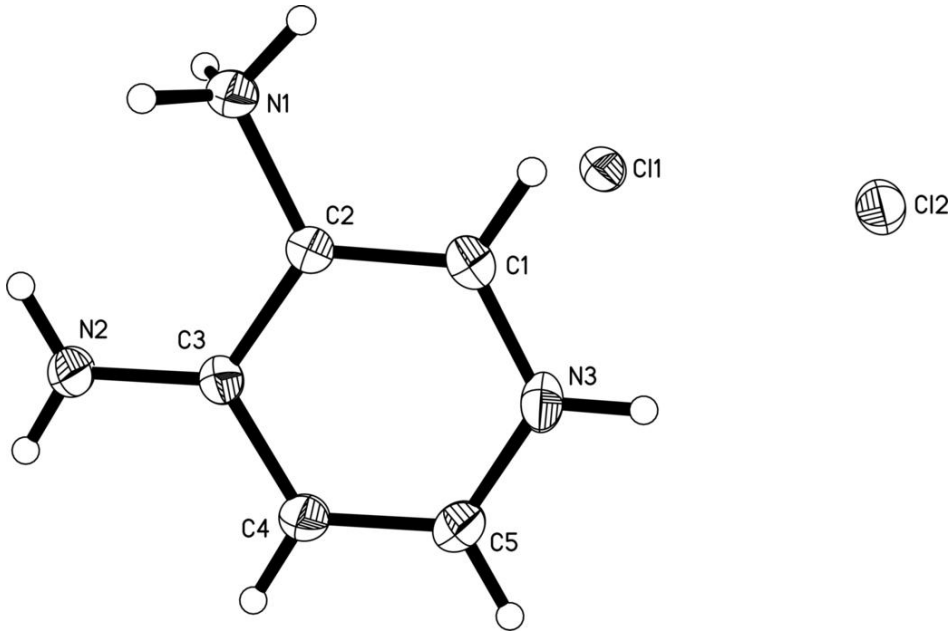

Fig. 2