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6-Methyl-2-[(6-methylpyridin-2-yl)amino]-pyridinium chloride chloroform monosolvate

Michaela Klass,* Christian Näther and Felix Tuczek

Institut für Anorganische Chemie, Christian-Albrechts-Universität zu Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany. *Correspondence e-mail: mklass@ac.uni-kiel.de

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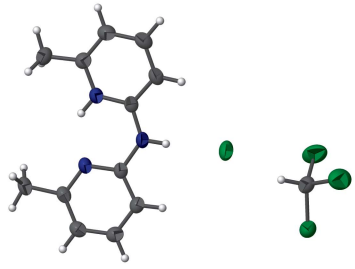
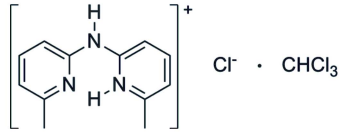
Structural data: full structural data are available from iucrdata.iucr.org

In the title solvated molecular salt, $C_{12}H_{14}N_3^+\cdot Cl^-\cdot CHCl_3$, the aromatic rings of the cation are nearly coplanar [dihedral angle = 6.30 (5)°] and an intramolecular N—H···N hydrogen bond occurs. In the crystal, the chloride ion accepts an N—H···Cl hydrogen bond from the cation and a C—H···Cl interaction from the solvent molecule. These trimeric units are linked by cation-to-anion C—H···Cl interactions into chains that propagate in the [001] direction.

3D view



Chemical scheme



Structure description

The title compound is shown in Fig. 1. The hydrogen bonding (Table 1) in the crystal structure is illustrated in Fig. 2.

For the related crystal structures of 2-(pyridin-1-ylamino)pyridinium iodide chloroform monosolvatesolvate, of 2-(pyridin-1-ylamino)pyridinium chloride dihydrate and of 2-(pyridin-1-ylamino)pyridinium chloride monohydrate, see: Chernychev *et al.* (2014) and Bock *et al.* (1998), respectively.

Synthesis and crystallization

Bis-(6-methylpyridin-2-yl)amine was synthesized according to the procedure given by Silberg *et al.* (2001). The compound was dissolved in dilute hydrochloric acid and extracted with dichloromethane. After solvent removal it was recrystallized from chloroform solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

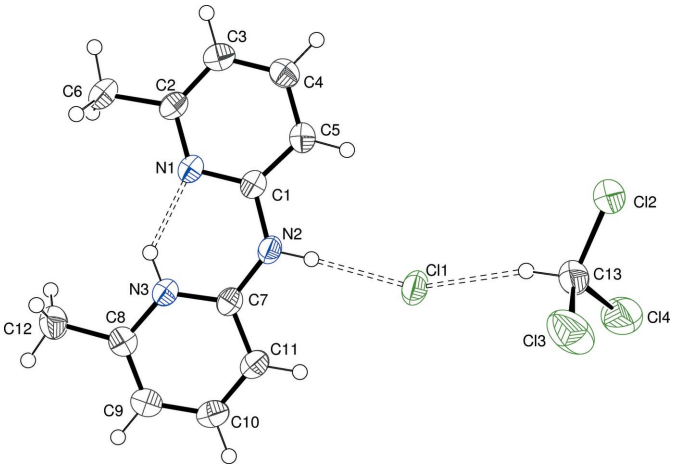


Figure 1
Part of the crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level. Hydrogen bonding is shown as dashed lines.

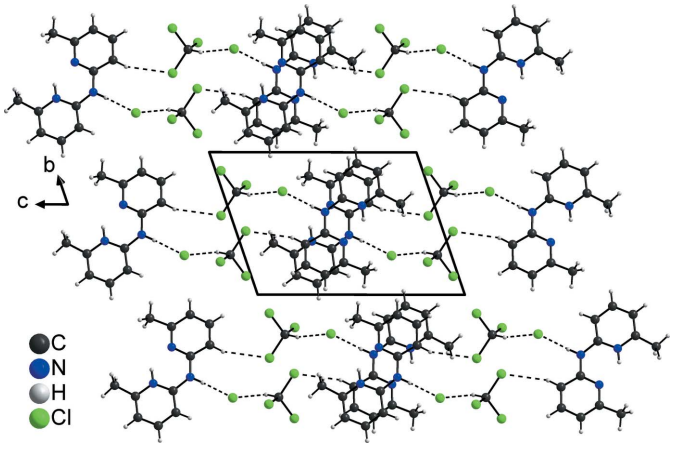


Figure 2
Crystal structure of the title compound with view along the crystallographic *a* axis. Hydrogen bonding is shown as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3n···N1	0.88	1.92	2.602 (2)	133
N2—H2*N*···Cl1	0.88	2.20	3.0771 (15)	177
C5—H5···Cl2^{i}	0.95	2.92	3.7969 (19)	154
C13—H13···Cl1	1.00	2.42	3.396 (2)	164

Symmetry code: (i) $-x+1, -y+1, -z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{12}N_3^+\cdot Cl^-\cdot CH_3Cl$
M_r	355.08
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	170
a, b, c (Å)	7.0474 (3), 9.7775 (5), 12.8695 (6)
α, β, γ (°)	71.744 (3), 89.177 (4), 72.582 (4)
V (Å^3)	800.44 (7)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.73
Crystal size (mm)	0.14 × 0.10 × 0.06
Data collection	
Diffractometer	Stoe IPDS2 diffractometer
Absorption correction	Numerical (*X-RED* and *X-SHAPE*; Stoe, 2008)
T_{min}, T_{max}	0.802, 0.941
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11648, 3487, 2885
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.100, 1.07
No. of reflections	3487
No. of parameters	184
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.50

Computer programs: *X-AREA* (Stoe, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 1999), *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x152467 [doi:10.1107/S2414314615024670]

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Crystal data

$C_{12}H_{12}N_3^+\cdot Cl^-\cdot CH_3Cl$
M_r = 355.08
Triclinic, $P\overline{1}$
a = 7.0474 (3) Å
b = 9.7775 (5) Å
c = 12.8695 (6) Å
α = 71.744 (3)°
β = 89.177 (4)°
γ = 72.582 (4)°
V = 800.44 (7) Å^3
Z = 2
$F(000)$ = 364
D_x = 1.473 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 11648 reflections
θ = 1.7–27.0°
μ = 0.73 mm^{-1}
T = 170 K
Block, colorless
0.14 × 0.10 × 0.06 mm

Data collection

Stoe IPDS-2 diffractometer
ω scan
Absorption correction: numerical (*X-RED* and *X-SHAPE*; Stoe, 2008)
T_{min} = 0.802, T_{max} = 0.941
11648 measured reflections
3487 independent reflections
2885 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.0°, θ_{min} = 1.7°
h = −9→9
k = −12→12
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.100
S = 1.07
3487 reflections
184 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.1904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (4)

Special details

Experimental. X-RED and X-SHAPE (STOE, 2008)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2170 (2)	0.56775 (18)	0.42537 (14)	0.0307 (3)
N1	0.1363 (2)	0.62988 (15)	0.50101 (12)	0.0311 (3)
C2	0.0123 (2)	0.77434 (18)	0.46826 (15)	0.0329 (4)
C3	−0.0301 (3)	0.85842 (19)	0.35861 (16)	0.0375 (4)
H3	−0.1169	0.9598	0.3365	0.045*
C4	0.0558 (3)	0.7931 (2)	0.28052 (16)	0.0382 (4)
H4	0.0282	0.8501	0.2046	0.046*
C5	0.1808 (3)	0.6459 (2)	0.31318 (15)	0.0347 (4)
H5	0.2401	0.5993	0.2610	0.042*
C6	−0.0711 (3)	0.8329 (2)	0.55911 (17)	0.0401 (4)
H6A	−0.1584	0.7759	0.5979	0.060*
H6B	−0.1480	0.9405	0.5284	0.060*
H6C	0.0386	0.8208	0.6106	0.060*
N2	0.3428 (2)	0.41937 (15)	0.46120 (12)	0.0324 (3)
H2N	0.3963	0.3812	0.4102	0.049*
C7	0.3893 (2)	0.32334 (18)	0.56621 (14)	0.0313 (3)
N3	0.3260 (2)	0.37459 (15)	0.64998 (12)	0.0318 (3)
H3N	0.2560	0.4706	0.6350	0.038*
C8	0.3655 (3)	0.2848 (2)	0.75685 (15)	0.0355 (4)
C9	0.4732 (3)	0.1354 (2)	0.78009 (16)	0.0398 (4)
H9	0.5021	0.0704	0.8541	0.048*
C10	0.5407 (3)	0.0785 (2)	0.69463 (17)	0.0403 (4)
H10	0.6145	−0.0255	0.7109	0.048*
C11	0.5014 (3)	0.17111 (19)	0.58816 (16)	0.0363 (4)
H11	0.5492	0.1331	0.5301	0.044*
C12	0.2855 (3)	0.3610 (2)	0.83920 (16)	0.0458 (4)
H12A	0.3130	0.2857	0.9129	0.069*
H12B	0.1410	0.4092	0.8227	0.069*
H12C	0.3498	0.4380	0.8362	0.069*
Cl1	0.53676 (8)	0.27483 (5)	0.28940 (4)	0.04733 (15)
C13	0.8018 (3)	0.2797 (2)	0.06653 (17)	0.0480 (5)
H13	0.7065	0.2978	0.1227	0.058*
Cl2	0.74696 (8)	0.44562 (6)	−0.04674 (4)	0.05151 (16)
Cl3	1.04500 (10)	0.23050 (10)	0.12560 (6)	0.0780 (2)
Cl4	0.77072 (12)	0.13078 (7)	0.02606 (6)	0.0723 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0285 (8)	0.0325 (8)	0.0352 (9)	−0.0113 (6)	0.0055 (7)	−0.0147 (7)
N1	0.0294 (7)	0.0312 (7)	0.0359 (7)	−0.0096 (5)	0.0065 (6)	−0.0154 (6)
C2	0.0285 (8)	0.0302 (8)	0.0432 (10)	−0.0098 (6)	0.0051 (7)	−0.0157 (7)
C3	0.0335 (9)	0.0324 (8)	0.0453 (10)	−0.0100 (7)	0.0010 (7)	−0.0111 (7)
C4	0.0387 (9)	0.0394 (9)	0.0355 (9)	−0.0142 (7)	0.0002 (7)	−0.0089 (7)
C5	0.0360 (9)	0.0390 (9)	0.0328 (9)	−0.0137 (7)	0.0062 (7)	−0.0147 (7)

C6	0.0390 (9)	0.0353 (9)	0.0512 (11)	−0.0103 (7)	0.0107 (8)	−0.0226 (8)
N2	0.0344 (7)	0.0325 (7)	0.0325 (7)	−0.0080 (6)	0.0078 (6)	−0.0160 (6)
C7	0.0281 (8)	0.0330 (8)	0.0365 (9)	−0.0108 (6)	0.0058 (7)	−0.0152 (7)
N3	0.0317 (7)	0.0306 (7)	0.0348 (7)	−0.0094 (5)	0.0061 (6)	−0.0133 (6)
C8	0.0327 (8)	0.0396 (9)	0.0360 (9)	−0.0145 (7)	0.0045 (7)	−0.0116 (7)
C9	0.0381 (9)	0.0383 (9)	0.0392 (10)	−0.0121 (7)	−0.0003 (8)	−0.0070 (8)
C10	0.0364 (9)	0.0323 (8)	0.0499 (11)	−0.0077 (7)	−0.0013 (8)	−0.0128 (8)
C11	0.0314 (8)	0.0346 (8)	0.0451 (10)	−0.0074 (7)	0.0024 (7)	−0.0186 (8)
C12	0.0497 (11)	0.0521 (11)	0.0367 (10)	−0.0149 (9)	0.0095 (8)	−0.0168 (9)
Cl1	0.0594 (3)	0.0437 (3)	0.0439 (3)	−0.0157 (2)	0.0212 (2)	−0.0220 (2)
C13	0.0480 (11)	0.0542 (11)	0.0368 (10)	−0.0102 (9)	0.0081 (8)	−0.0135 (9)
Cl2	0.0574 (3)	0.0499 (3)	0.0397 (3)	−0.0087 (2)	0.0098 (2)	−0.0119 (2)
Cl3	0.0540 (4)	0.1047 (5)	0.0553 (4)	−0.0188 (3)	−0.0076 (3)	−0.0035 (3)
Cl4	0.0938 (5)	0.0535 (3)	0.0663 (4)	−0.0184 (3)	0.0004 (4)	−0.0188 (3)

Geometric parameters (Å, º)

C1—N1	1.337 (2)	C7—C11	1.399 (2)
C1—N2	1.388 (2)	N3—C8	1.359 (2)
C1—C5	1.391 (2)	N3—H3N	0.8800
N1—C2	1.355 (2)	C8—C9	1.366 (3)
C2—C3	1.376 (3)	C8—C12	1.491 (3)
C2—C6	1.496 (3)	C9—C10	1.398 (3)
C3—C4	1.392 (3)	C9—H9	0.9500
C3—H3	0.9500	C10—C11	1.363 (3)
C4—C5	1.377 (3)	C10—H10	0.9500
C4—H4	0.9500	C11—H11	0.9500
C5—H5	0.9500	C12—H12A	0.9800
C6—H6A	0.9800	C12—H12B	0.9800
C6—H6B	0.9800	C12—H12C	0.9800
C6—H6C	0.9800	C13—Cl3	1.749 (2)
N2—C7	1.360 (2)	C13—Cl2	1.751 (2)
N2—H2N	0.8800	C13—Cl4	1.763 (2)
C7—N3	1.343 (2)	C13—H13	1.0000
N1—C1—N2	118.11 (15)	C7—N3—C8	122.93 (15)
N1—C1—C5	122.75 (15)	C7—N3—H3N	118.5
N2—C1—C5	119.14 (15)	C8—N3—H3N	118.5
C1—N1—C2	119.32 (15)	N3—C8—C9	118.58 (17)
N1—C2—C3	121.07 (16)	N3—C8—C12	115.71 (16)
N1—C2—C6	115.17 (16)	C9—C8—C12	125.71 (18)
C3—C2—C6	123.76 (16)	C8—C9—C10	119.86 (18)
C2—C3—C4	119.17 (16)	C8—C9—H9	120.1
C2—C3—H3	120.4	C10—C9—H9	120.1
C4—C3—H3	120.4	C11—C10—C9	120.55 (17)
C5—C4—C3	120.08 (17)	C11—C10—H10	119.7
C5—C4—H4	120.0	C9—C10—H10	119.7
C3—C4—H4	120.0	C10—C11—C7	118.61 (17)

C4—C5—C1	117.60 (16)	C10—C11—H11	120.7
C4—C5—H5	121.2	C7—C11—H11	120.7
C1—C5—H5	121.2	C8—C12—H12A	109.5
C2—C6—H6A	109.5	C8—C12—H12B	109.5
C2—C6—H6B	109.5	H12A—C12—H12B	109.5
H6A—C6—H6B	109.5	C8—C12—H12C	109.5
C2—C6—H6C	109.5	H12A—C12—H12C	109.5
H6A—C6—H6C	109.5	H12B—C12—H12C	109.5
H6B—C6—H6C	109.5	Cl3—C13—Cl2	111.00 (12)
C7—N2—C1	127.70 (14)	Cl3—C13—Cl4	110.30 (12)
C7—N2—H2N	115.6	Cl2—C13—Cl4	109.85 (12)
C1—N2—H2N	116.7	Cl3—C13—H13	108.5
N3—C7—N2	119.76 (15)	Cl2—C13—H13	108.5
N3—C7—C11	119.47 (16)	Cl4—C13—H13	108.5
N2—C7—C11	120.77 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3n···N1	0.88	1.92	2.602 (2)	133
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