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4-(2-Pyridyl)piperazin-1-ium chloride

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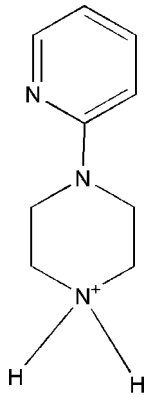
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.061; data-to-parameter ratio = 12.2.

The title compound, $C_9H_{14}N_3^+\cdot Cl^-$, crystallizes at room temperature from chloroform as a zwitterion. The molecules are linked by N—H···Cl hydrogen bonds into chains. The piperazine ring adopts a chair conformation.

Related literature

For related literature, see: The Merck Index (1989).



Experimental

Crystal data

$C_9H_{14}N_3^+\cdot Cl^-$
M_r = 199.68
Orthorhombic, $P2_12_12_1$
a = 7.1757 (6) Å
b = 7.2196 (6) Å
c = 18.7629 (16) Å
V = 972.02 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 100 (2) K
0.40 × 0.25 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
5613 measured reflections
1956 independent reflections
1781 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.061
S = 0.96
1956 reflections
160 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 787 Freidel pairs
Flack parameter: 0.04 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···Cl1	0.979 (19)	2.15 (2)	3.1020 (17)	164.3 (15)
N1—H1···Cl1^{i}	0.91 (2)	2.19 (2)	3.0961 (17)	174.3 (17)

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); 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: AT2451).

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  doi:10.1107/S1600536807053470

supplementary materials

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4-(2-Pyridyl)piperazin-1-ium chloride

Sadiq-ur-Rehman, S. Saeed, S. Ali, S. Shahzadi and M. Helliwell

Comment

Piperazine is freely soluble in water and ethylene glycol, but insoluble in diethyl ether. It is a weak base with a pKb of 4.19; the pH of a 10% aqueous solution is 10.8–11.8. A large number of piperazine compounds have anthelmintic action. Piperazines are also used in the manufacture of plastics, resins, pesticides, brake fluid and other industrial materials (The Merck Index, 1989). The structure of (I) is shown in Fig. 1.

The N—C distances in piperazin-4-ium ring are 1.485 (3) and 1.490 (2) Å, while in the pyridine ring the N—C distance are 1.345 (2) Å. Hydrogen bonds of the type N—H···Cl link the molecules into chains, with N···Cl separation of 3.0961 (17) and 3.1020 (17) Å (Fig. 2).

Experimental

1-(2-Pyridinyl)piperazine (1 mmol) and trimethyltin chloride (1 mmol) were suspended in dry chloroform (150 ml) in a round bottom two necked flask. The mixture was stirred at room temperature. Colourless crystals of the title compound obtained accidently after recrystallization in acetone. (Yield 70%°; m.p. 393 K).

Refinement

H atoms were included in difference map positions and refined freely, with C—H distances ranging from 0.84 (2) − 1.00 (2) Å and N—H distances of 0.91 (2) and 0.98 (2) Å.

Figures

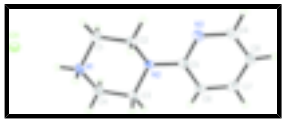

Fig. 1. Structure of (I) with displacement ellipsoids drawn at the 50% probability level.

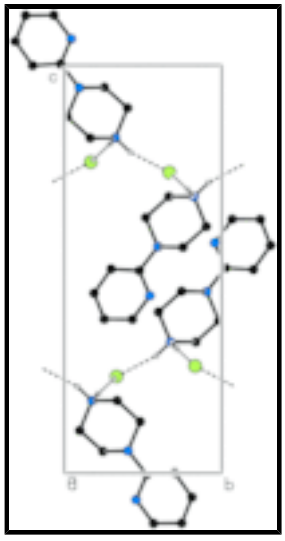

Fig. 2. Packing of (I) viewed down a showing the N—H···Cl hydrogen bonding. H atoms not involved in hydrogen bonding have been omitted for clarity.

4-(2-Pyridyl)piperazin-1-ium chloride

Crystal data

$C_9H_{14}ClN_3$	F_{000} = 424
M_r = 199.68	D_x = 1.364 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 2404 reflections
a = 7.1757 (6) Å	θ = 3.0–26.3º
b = 7.2196 (6) Å	μ = 0.35 mm^{-1}
c = 18.7629 (16) Å	T = 100 (2) K
V = 972.02 (14) Å^3	Plate, colourless
Z = 4	0.40 × 0.25 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1781 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.044
Monochromator: graphite	θ_{max} = 26.4º
T = 100(2) K	θ_{min} = 2.2º
φ and ω scans	h = −8→8
Absorption correction: none	k = −9→8
5613 measured reflections	l = −20→23
1956 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	Only H-atom coordinates refined

$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.96$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
1956 reflections	$\Delta\rho_{min} = -0.16$ e Å^{-3}
160 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 787 Freidel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.04 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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^ > 2sigma(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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.24771 (7)	0.33171 (5)	0.23724 (2)	0.02020 (12)
N1	0.3814 (2)	0.6758 (2)	0.32203 (8)	0.0201 (4)
N2	0.3397 (2)	0.90492 (19)	0.44477 (7)	0.0157 (3)
N3	0.4096 (2)	0.9562 (2)	0.56481 (8)	0.0194 (3)
C1	0.4004 (3)	0.5979 (3)	0.39523 (10)	0.0195 (4)
C2	0.4664 (3)	0.7455 (3)	0.44616 (10)	0.0178 (4)
C3	0.3159 (3)	0.9827 (3)	0.37348 (9)	0.0186 (4)
C4	0.2510 (3)	0.8355 (3)	0.32164 (9)	0.0221 (4)
C5	0.3404 (3)	1.0234 (2)	0.50341 (9)	0.0150 (4)
C6	0.2672 (3)	1.2021 (2)	0.49926 (9)	0.0174 (4)
C7	0.2704 (3)	1.3113 (2)	0.55864 (9)	0.0218 (4)
C8	0.3435 (3)	1.2463 (3)	0.62216 (10)	0.0201 (4)
C9	0.4074 (3)	1.0673 (3)	0.62279 (9)	0.0202 (4)
H1	0.494 (3)	0.714 (3)	0.3056 (9)	0.024*
H2	0.336 (3)	0.584 (3)	0.2878 (9)	0.024*
H3	0.485 (3)	0.500 (3)	0.3933 (9)	0.024*
H4	0.274 (3)	0.553 (2)	0.4082 (8)	0.024*
H5	0.595 (3)	0.784 (2)	0.4338 (9)	0.024*
H6	0.474 (3)	0.697 (2)	0.4932 (10)	0.024*
H7	0.219 (3)	1.082 (3)	0.3732 (9)	0.024*
H8	0.431 (3)	1.036 (3)	0.3556 (9)	0.024*
H9	0.244 (3)	0.885 (2)	0.2767 (9)	0.024*
H10	0.129 (3)	0.795 (3)	0.3334 (9)	0.024*

H11	0.217 (3)	1.237 (3)	0.4612 (9)	0.024*
H12	0.223 (3)	1.440 (2)	0.5546 (9)	0.024*
H13	0.350 (3)	1.317 (3)	0.6642 (9)	0.024*
H14	0.453 (3)	1.016 (3)	0.6682 (9)	0.024*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0216 (2)	0.0179 (2)	0.0211 (2)	−0.0011 (2)	0.0015 (2)	−0.00328 (17)
N1	0.0234 (9)	0.0198 (8)	0.0171 (8)	−0.0057 (8)	0.0008 (7)	−0.0045 (7)
N2	0.0198 (8)	0.0144 (7)	0.0129 (7)	0.0023 (7)	−0.0015 (6)	0.0020 (6)
N3	0.0203 (8)	0.0213 (8)	0.0167 (8)	−0.0004 (7)	−0.0005 (6)	−0.0002 (7)
C1	0.0193 (10)	0.0171 (10)	0.0222 (10)	−0.0004 (8)	0.0002 (9)	−0.0002 (8)
C2	0.0198 (11)	0.0171 (9)	0.0165 (9)	0.0031 (8)	−0.0025 (8)	0.0015 (8)
C3	0.0229 (10)	0.0181 (9)	0.0149 (9)	0.0010 (8)	−0.0030 (8)	0.0015 (8)
C4	0.0253 (10)	0.0245 (9)	0.0164 (8)	−0.0004 (13)	−0.0044 (10)	0.0020 (8)
C5	0.0125 (9)	0.0167 (9)	0.0158 (9)	−0.0015 (8)	0.0024 (7)	0.0025 (8)
C6	0.0179 (10)	0.0170 (9)	0.0172 (8)	0.0007 (9)	−0.0012 (9)	0.0042 (7)
C7	0.0198 (11)	0.0159 (9)	0.0297 (10)	0.0004 (9)	0.0028 (9)	0.0016 (8)
C8	0.0189 (10)	0.0222 (10)	0.0191 (10)	−0.0035 (9)	0.0039 (8)	−0.0062 (8)
C9	0.0185 (10)	0.0277 (11)	0.0143 (9)	−0.0012 (8)	−0.0003 (8)	0.0027 (8)

Geometric parameters (Å, °)

N1—C4	1.485 (3)	C3—C4	1.514 (2)
N1—C1	1.490 (2)	C3—H7	0.998 (19)
N1—H1	0.91 (2)	C3—H8	0.97 (2)
N1—H2	0.979 (19)	C4—H9	0.918 (17)
N2—C5	1.394 (2)	C4—H10	0.95 (2)
N2—C3	1.461 (2)	C5—C6	1.395 (2)
N2—C2	1.467 (2)	C6—C7	1.365 (2)
N3—C5	1.345 (2)	C6—H11	0.840 (19)
N3—C9	1.352 (2)	C7—C8	1.384 (3)
C1—C2	1.508 (3)	C7—H12	0.991 (19)
C1—H3	0.93 (2)	C8—C9	1.371 (3)
C1—H4	1.00 (2)	C8—H13	0.939 (18)
C2—H5	0.99 (2)	C9—H14	0.985 (17)
C2—H6	0.951 (18)		
C4—N1—C1	110.81 (14)	N2—C3—H8	111.7 (11)
C4—N1—H1	108.9 (12)	C4—C3—H8	108.5 (11)
C1—N1—H1	110.2 (11)	H7—C3—H8	107.6 (15)
C4—N1—H2	108.4 (11)	N1—C4—C3	110.37 (16)
C1—N1—H2	112.2 (11)	N1—C4—H9	110.1 (12)
H1—N1—H2	106.2 (15)	C3—C4—H9	109.3 (11)
C5—N2—C3	119.17 (14)	N1—C4—H10	109.8 (12)
C5—N2—C2	117.74 (14)	C3—C4—H10	110.6 (11)
C3—N2—C2	112.98 (14)	H9—C4—H10	106.5 (17)
C5—N3—C9	118.09 (15)	N3—C5—N2	117.13 (15)

N1—C1—C2	110.24 (15)	N3—C5—C6	121.36 (15)
N1—C1—H3	108.1 (11)	N2—C5—C6	121.48 (15)
C2—C1—H3	110.9 (12)	C7—C6—C5	118.83 (16)
N1—C1—H4	105.4 (10)	C7—C6—H11	121.8 (13)
C2—C1—H4	111.3 (9)	C5—C6—H11	119.2 (13)
H3—C1—H4	110.7 (16)	C6—C7—C8	120.90 (17)
N2—C2—C1	110.42 (15)	C6—C7—H12	118.2 (10)
N2—C2—H5	110.6 (11)	C8—C7—H12	120.9 (10)
C1—C2—H5	110.0 (11)	C9—C8—C7	116.97 (17)
N2—C2—H6	110.0 (11)	C9—C8—H13	119.1 (12)
C1—C2—H6	110.3 (10)	C7—C8—H13	123.9 (12)
H5—C2—H6	105.4 (16)	N3—C9—C8	123.80 (17)
N2—C3—C4	110.76 (15)	N3—C9—H14	118.1 (11)
N2—C3—H7	111.3 (10)	C8—C9—H14	118.1 (11)
C4—C3—H7	106.7 (10)		
C4—N1—C1—C2	−57.5 (2)	C3—N2—C5—N3	−163.18 (16)
C5—N2—C2—C1	158.72 (16)	C2—N2—C5—N3	−20.2 (2)
C3—N2—C2—C1	−56.1 (2)	C3—N2—C5—C6	18.7 (3)
N1—C1—C2—N2	56.1 (2)	C2—N2—C5—C6	161.74 (17)
C5—N2—C3—C4	−159.81 (17)	N3—C5—C6—C7	1.5 (3)
C2—N2—C3—C4	55.5 (2)	N2—C5—C6—C7	179.50 (18)
C1—N1—C4—C3	56.9 (2)	C5—C6—C7—C8	−0.5 (3)
N2—C3—C4—N1	−55.2 (2)	C6—C7—C8—C9	−1.5 (3)
C9—N3—C5—N2	−178.43 (16)	C5—N3—C9—C8	−1.9 (3)
C9—N3—C5—C6	−0.3 (3)	C7—C8—C9—N3	2.8 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···Cl1	0.979 (19)	2.15 (2)	3.1020 (17)	164.3 (15)
N1—H1···Cl1^{i}	0.91 (2)	2.19 (2)	3.0961 (17)	174.3 (17)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1

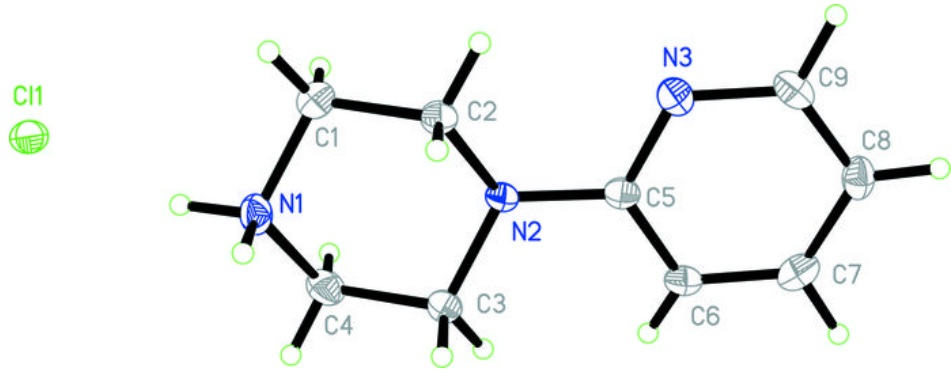

Fig. 2