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Crystal structure of 2-(1*H*-imidazol-4-yl)-ethanaminium chloride

Imene Belfilali,[a] Siham Yebdri,[a] Samira Louhibi,[a]* Leila Boukli-hacene[a] and Thierry Roisnel[b]

[a]Laboratoire de Chimie Inorganique et Environnement, University of Tlemcen, BP 119, 13000, Tlemcen, Algeria, and [b]Centre de Diffractometrie X, UMR 6226 CNRS, Unite Sciences Chimiques de Rennes, Universite de Rennes I, 263 Avenue du General Leclerc, 35042 Rennes, France. *Correspondence e-mail: samhibi1@yahoo.fr

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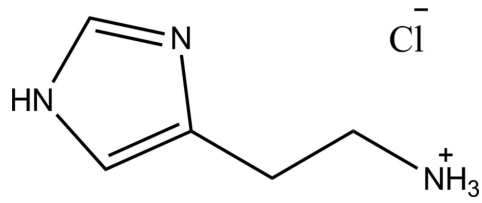
The title molecular salt, $C_5H_{10}N_3^+\cdot Cl^-$, was obtained as by-product in the attempted synthesis of a histamine derivative. The terminal amino group of the starting material is protonated. The $C_{imidazole}-C-C-N(H_3)^+$ group in the cation is in an *anti* conformation with a torsion angle of 176.22 (10)°. In the crystal, cations and anions are linked *via* N—H···N and N—H—Cl hydrogen bonds, forming a two-dimensional network parallel to $(10\bar{1})$. A single weak C—H···Cl hydrogen bond completes a three-dimensional network.

Keywords: crystal structure; histamine; imidazole; chloride Ion; protonation; hydrogen bonding.

CCDC reference: 1051527

1. Related literature

For the biological and pharmacological applications of histamine derivatives, see: Barnes *et al.* (2001); Schwartz *et al.* (1991); Bachert *et al.* (1998); Emanuel *et al.* (1999); Apáti *et al.* (2012). For a study of a histamine copper(II) chloride complex, see: Belfilali *et al.* (2015). For the general chemistry of transition metal ions with histamine, see: Mikulski *et al.* (2012); Kowalik-Jankowska *et al.* (2010); Selmeczi *et al.* (2012). For a related structure, see: Prout *et al.* (1974).



2. Experimental

2.1. Crystal data

$C_5H_{10}N_3^+\cdot Cl^-$
M_r = 147.61
Monoclinic, $P2_1/n$
a = 4.5840 (2) Å
b = 9.1614 (3) Å
c = 17.3114 (5) Å
β = 91.682 (1)°
V = 726.69 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 150 K
0.41 × 0.13 × 0.08 mm

2.2. Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)'
T_{min} = 0.868, T_{max} = 0.965
5568 measured reflections
1645 independent reflections
1494 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.08
1645 reflections
86 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N5^{i}	0.91	1.96	2.8508 (15)	168
N1—H1B···Cl1^{i}	0.91	2.28	3.1557 (11)	160
N1—H1C···Cl1^{ii}	0.91	2.39	3.2443 (11)	157
N7—H7···Cl1^{iii}	0.78 (2)	2.40 (2)	3.1645 (12)	168 (2)
C2—H2A···Cl1^{iv}	0.99	2.72	3.6974 (14)	168

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x, y+1, z$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x+1, y+1, z$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012) and *CRYSCAL* (T. Roisnel, local program).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: LH5756).

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Crystal structure of 2-(1*H*-imidazol-4-yl)ethanaminium chloride

Imene Belfilali, Siham Yebdri, Samira Louhibi, Leila Boukli-hacene and Thierry Roisnel

S1. Structural commentary

Histamine (2-(1H-imidazol-4-yl)ethanamine) is a biogenic amine present in essentially all mammalian tissues and involved in several defense mechanisms of the body. It plays a role in various physiological processes, such as control of gastric acid secretion, neurotransmission, regulation of the microcirculation, and modulation of inflammatory (Barnes *et al.*, 2001) and immunological reactions (Schwartz *et al.*, 1991; Bachert *et al.*, 1998; Emanuel *et al.*, 1999) as well as its uses in pharmacology (Apáti *et al.*, 2012). Moreover, the interaction of transition metal ions with histamine (Mikulski *et al.*, 2012), play a key role in catalysis processes (Kowalik-Jankowska *et al.*, 2010; Selmeczi *et al.*, 2012). We have previously reported the preparation and the crystal structure of the histamine copper(II) chloride complex and its catalytic activity study (Belfilali *et al.*, 2015). In this study, we report the synthesis and crystal structure determination of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The organic cation displays a trans conformation with respect to the amine group and the imidazole ring about the –CH_2—CH_2– bond of the side chain with a torsion angle of 176.22 (10)° for N1–C2–C3–C4. The bond lengths and angles are within normal ranges and are comparable to a related structure (Prout *et al.*, 1974). In the crystal, cations and anions are linked via N—H···N and N—H—Cl hydrogen bonds two form a two-dimensional network (Fig. 2) parallel to (10$\bar{1}$). A single weak C—H···Cl hydrogen bond completes a three-dimensinal network.

S2. Synthesis and crystallization

A mixture of histamine dihydrochloride (1.0 mmol) and methyl-1hydroxy-2-naphthoate (1 mmol) were taken in a beaker placed in a microwave oven and irradiated at 200 watt for 5 minutes. After completion the reaction, the reaction mixture was allowed to reach room temperature and the resulting crystals were separated by filtration.

S3. Refinement

H atoms bonded to C atoms were included in calculated positions with C—H = 0.95 – 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to N1 were included in calculated positions with N—H = 0.91Å and U_{iso}(H) = 1.5U_{eq}(N). The H atom bonded to N7 was refined independently with an isotropic displacement parameter.

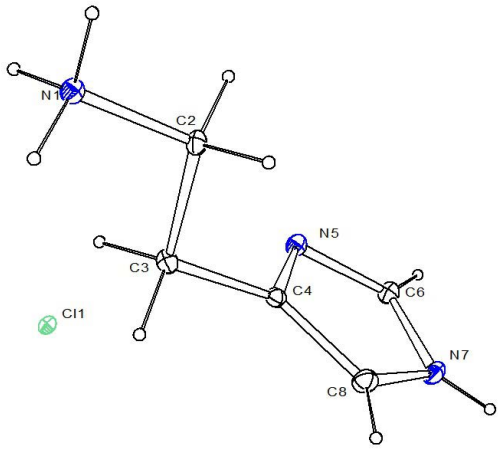

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

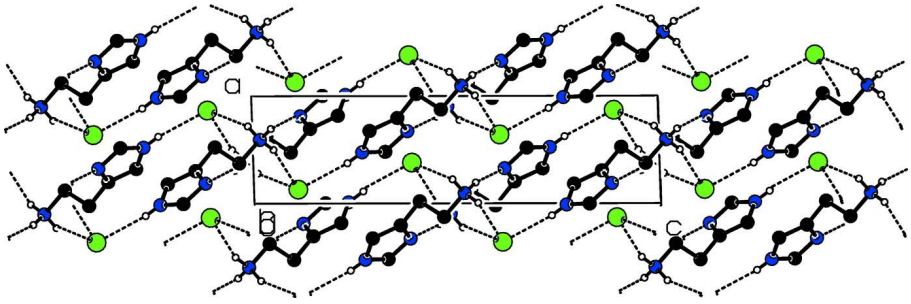

Figure 2

Part of the crystal structure with hydrogen bonds shown as dashed lines.

2-(1*H*-Imidazol-4-yl)ethanaminium chloride

Crystal data

$C_5H_{10}N_3^+\cdot Cl^-$
$M_r = 147.61$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.5840\ (2)$ Å
$b = 9.1614\ (3)$ Å
$c = 17.3114\ (5)$ Å
$\beta = 91.682\ (1)°$
$V = 726.69\ (4)$ Å^3
$Z = 4$
$F(000) = 312$
$D_x = 1.349$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2978 reflections
$\theta = 4.6–27.5°$
$\mu = 0.44$ mm^{-1}
$T = 150$ K
Prism, colourless
0.41 × 0.13 × 0.08 mm

Data collection

Bruker APEXII diffractometer
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)′
$T_{min} = 0.868$, $T_{max} = 0.965$
5568 measured reflections
1645 independent reflections
1494 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$

$h = -5 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.076$
$S = 1.08$
1645 reflections
86 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
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.246P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4106 (2)	0.80020 (12)	−0.01832 (6)	0.0174 (2)
H1A	0.3108	0.7313	−0.0462	0.026*
H1B	0.5029	0.8614	−0.051	0.026*
H1C	0.2838	0.8521	0.0104	0.026*
C2	0.6308 (3)	0.72762 (15)	0.03395 (7)	0.0174 (3)
H2A	0.7497	0.8028	0.0613	0.021*
H2B	0.7631	0.6675	0.0029	0.021*
C3	0.4835 (3)	0.63146 (15)	0.09277 (8)	0.0192 (3)
H3A	0.3615	0.6926	0.1262	0.023*
H3B	0.3543	0.5605	0.0655	0.023*
C4	0.7051 (3)	0.55095 (15)	0.14180 (7)	0.0169 (3)
N5	0.8253 (2)	0.41984 (12)	0.11844 (6)	0.0184 (2)
C6	1.0162 (3)	0.38371 (15)	0.17436 (7)	0.0197 (3)
H6	1.1334	0.2982	0.1741	0.024*
N7	1.0229 (3)	0.48288 (14)	0.23111 (7)	0.0223 (3)
H7	1.124 (5)	0.481 (2)	0.2682 (14)	0.05*
C8	0.8270 (3)	0.58985 (16)	0.21139 (8)	0.0229 (3)
H8	0.7842	0.6747	0.2405	0.027*
Cl1	0.13058 (7)	0.01456 (3)	0.108958 (17)	0.01841 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0185 (5)	0.0155 (6)	0.0180 (5)	−0.0019 (4)	−0.0008 (4)	−0.0004 (4)
C2	0.0152 (6)	0.0180 (6)	0.0190 (6)	−0.0021 (5)	−0.0022 (5)	−0.0002 (5)
C3	0.0156 (6)	0.0200 (7)	0.0220 (7)	−0.0008 (5)	0.0007 (5)	0.0009 (5)
C4	0.0160 (6)	0.0180 (6)	0.0169 (6)	−0.0017 (5)	0.0031 (5)	0.0006 (5)
N5	0.0206 (5)	0.0155 (5)	0.0189 (5)	−0.0016 (4)	−0.0018 (4)	−0.0006 (5)
C6	0.0218 (6)	0.0176 (6)	0.0197 (6)	−0.0007 (5)	−0.0012 (5)	0.0015 (5)
N7	0.0238 (6)	0.0268 (6)	0.0161 (6)	0.0001 (5)	−0.0041 (5)	−0.0009 (5)
C8	0.0252 (7)	0.0233 (7)	0.0202 (7)	0.0034 (6)	0.0008 (5)	−0.0046 (6)
Cl1	0.02061 (18)	0.01856 (18)	0.01589 (19)	−0.00105 (12)	−0.00236 (12)	−0.00087 (11)

Geometric parameters (Å, º)

N1—C2	1.4920 (16)	C3—H3B	0.99
N1—H1A	0.91	C4—C8	1.3604 (18)
N1—H1B	0.91	C4—N5	1.3866 (17)
N1—H1C	0.91	N5—C6	1.3277 (16)
C2—C3	1.5196 (18)	C6—N7	1.3379 (18)
C2—H2A	0.99	C6—H6	0.95
C2—H2B	0.99	N7—C8	1.3658 (18)
C3—C4	1.4985 (18)	N7—H7	0.78 (2)
C3—H3A	0.99	C8—H8	0.95
C2—N1—H1A	109.5	C2—C3—H3B	109.4
C2—N1—H1B	109.5	H3A—C3—H3B	108
H1A—N1—H1B	109.5	C8—C4—N5	109.20 (11)
C2—N1—H1C	109.5	C8—C4—C3	128.79 (13)
H1A—N1—H1C	109.5	N5—C4—C3	121.99 (11)
H1B—N1—H1C	109.5	C6—N5—C4	105.21 (11)
N1—C2—C3	111.03 (10)	N5—C6—N7	111.50 (12)
N1—C2—H2A	109.4	N5—C6—H6	124.2
C3—C2—H2A	109.4	N7—C6—H6	124.2
N1—C2—H2B	109.4	C6—N7—C8	107.63 (11)
C3—C2—H2B	109.4	C6—N7—H7	126.3 (16)
H2A—C2—H2B	108	C8—N7—H7	126.1 (16)
C4—C3—C2	110.96 (10)	C4—C8—N7	106.46 (12)
C4—C3—H3A	109.4	C4—C8—H8	126.8
C2—C3—H3A	109.4	N7—C8—H8	126.8
C4—C3—H3B	109.4		
N1—C2—C3—C4	176.22 (10)	C4—N5—C6—N7	0.21 (15)
C2—C3—C4—C8	93.03 (17)	N5—C6—N7—C8	−0.18 (16)
C2—C3—C4—N5	−84.87 (15)	N5—C4—C8—N7	0.06 (15)
C8—C4—N5—C6	−0.16 (15)	C3—C4—C8—N7	−178.06 (12)
C3—C4—N5—C6	178.11 (12)	C6—N7—C8—C4	0.07 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N5^{i}	0.91	1.96	2.8508 (15)	168
N1—H1*B*···Cl1^{i}	0.91	2.28	3.1557 (11)	160
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C2—H2*A*···Cl1^{iv}	0.99	2.72	3.6974 (14)	168

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) *x*, *y*+1, *z*; (iii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iv) *x*+1, *y*+1, *z*.