

2,3-Dimethyl-1*H*-imidazol-3-ium chloride

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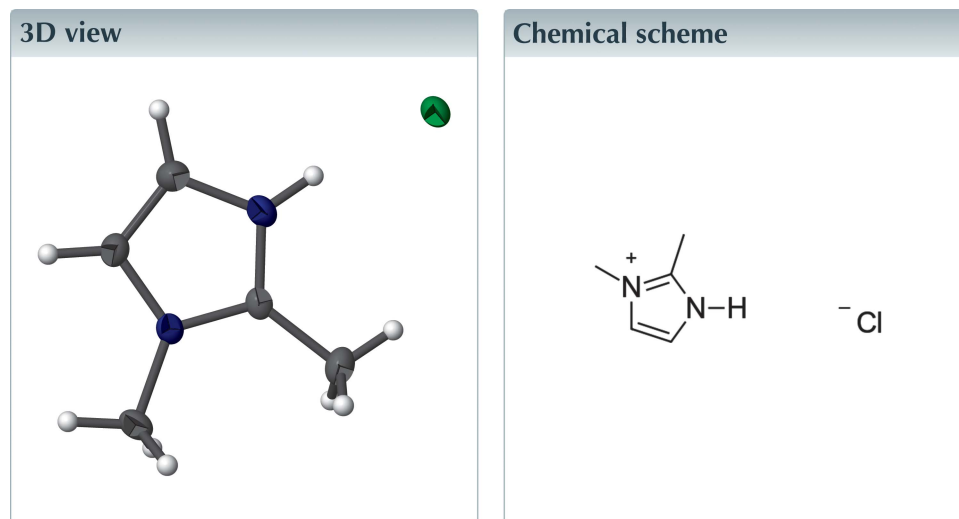
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Structural data: full structural data are available from iucrdata.iucr.org

The title salt, $C_5H_9N_2^+ \cdot Cl^-$, exhibits multiple hydrogen-bonding interactions between the cationic imidazole moiety and the chloride anion. The protonated aromatic nitrogen moiety displays the shortest hydrogen-bonding interactions while weaker hydrogen bonding is observed between the aromatic H atoms and the chloride anion. The crystal studied was refined as a two-component inversion twin with a twin ratio of 0.71 (5) to 0.29 (5).



Structure description

The title structure, 2,3-dimethyl-1*H*-imidazol-3-ium chloride (Fig. 1), crystallizes in the $P2_12_12_1$ orthorhombic space group with a single cation–anion pair in the asymmetric unit. The acidic hydrogen, H1, exhibits a strong hydrogen bond to the chloride anion with a distance of 2.122 (19) Å. Longer hydrogen bonds between the chloride anion and H atoms on both methyl groups on the imidazolium ring as well as to the aromatic H atoms on adjacent cations form the dominant intermolecular interactions in the overall network (see Table 1). The positioning of the cations, likely to facilitate hydrogen bonding, also precludes any possible long-distance π – π interactions given the canted angles of the rings with respect to each other (see Fig. 2).

Synthesis and crystallization

1,2-Dimethylimidazole (0.2568 g, 2.662 mmol) and trityl chloride (0.7439 g, 2.668 mmol) were dissolved in separate 50 mL beakers with toluene. The reactants were then combined in a single-necked 100 mL round-bottom flask equipped with a magnetic stir bar and left to stir for 2 d at room temperature. The solvent was removed under vacuum leaving a white solid residue. This solid was washed twice with tetrahydrofuran and

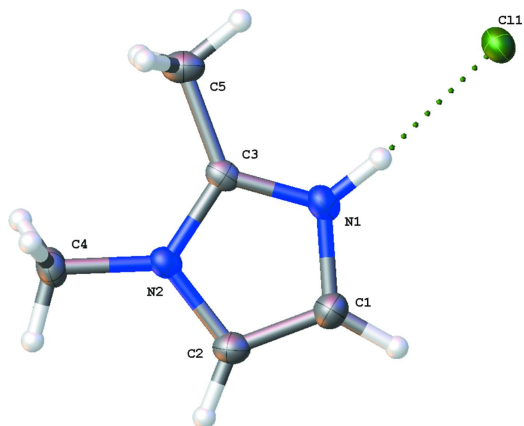


Figure 1
The title compound shown with 50% probability ellipsoids. Carbon (grey), hydrogen (white), nitrogen (blue), chlorine (green).

recovered *via* vacuum filtration. Crystals were grown at room temperature by vapor diffusion with acetonitrile as the solvent and tetrahydrofuran as the anti-solvent. Colorless crystals of the hydrolyzed byproduct (2,3-dimethyl-1*H*-imidazol-3-ium chloride) were observed within one week.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was refined as a

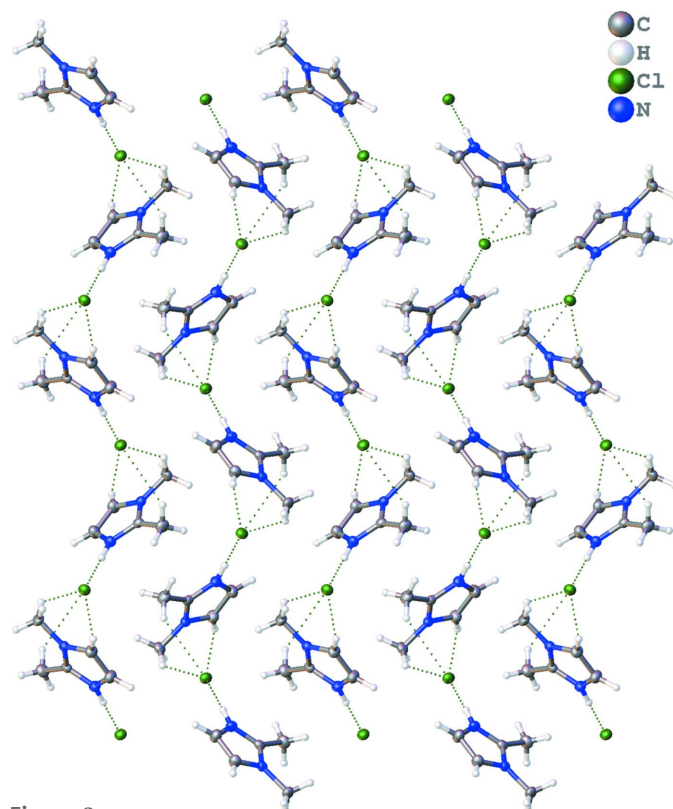


Figure 2
Packing diagram of the title compound showing a zigzag network of ion pairs held together through hydrogen bonds.

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1···Cl1	0.923 (19)	2.122 (19)	3.0396 (10)	172.3 (17)
C2—H2···Cl1 ⁱ	0.955 (17)	2.695 (17)	3.6084 (11)	160.2 (13)
C4—H4C···Cl1 ⁱⁱ	1.005 (19)	2.85 (2)	3.6601 (12)	138.3 (14)
C5—H5A···Cl1 ⁱⁱ	0.94 (2)	2.887 (19)	3.6415 (13)	138.0 (14)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₅ H ₉ N ₂ ⁺ ·Cl ⁻
<i>M_r</i>	132.59
Crystal system, space group	Orthorhombic, <i>P</i> ₂ ₁ ₂ ₁
Temperature (K)	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.3076 (4), 9.5490 (6), 11.3951 (8)
<i>V</i> (Å ³)	686.34 (8)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.46
Crystal size (mm)	0.53 × 0.49 × 0.42
Data collection	
Diffractometer	Bruker AXS D8 Quest CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.713, 0.747
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	13899, 2602, 2499
<i>R</i> _{int}	0.029
(sin θ/λ) _{max} (Å ⁻¹)	0.768
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.019, 0.050, 1.11
No. of reflections	2602
No. of parameters	111
H-atom treatment	All H-atom parameters refined
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.21, -0.15
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.29 (5)

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2018), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), *shelXle* (Hübschle *et al.*, 2011), *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010).

two-component inversion twin with a twin ratio of 0.71 (5) to 0.29 (5).

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full crystallographic data

IUCrData (2020). 5, x200660 [https://doi.org/10.1107/S2414314620006604]

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2,3-Dimethyl-1*H*-imidazol-3-ium chloride*Crystal data*

$C_5H_9N_2^+ \cdot Cl^-$

$M_r = 132.59$

Orthorhombic, $P2_12_12_1$

$a = 6.3076$ (4) Å

$b = 9.5490$ (6) Å

$c = 11.3951$ (8) Å

$V = 686.34$ (8) Å³

$Z = 4$

$F(000) = 280$

$D_x = 1.283$ Mg m⁻³

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

Cell parameters from 9928 reflections

$\theta = 2.8$ – 33.1°

$\mu = 0.46$ mm⁻¹

$T = 150$ K

Prism, colourless

$0.53 \times 0.49 \times 0.42$ mm

Data collection

Bruker AXS D8 Quest CMOS

diffractometer

Detector resolution: 10.4167 pixels mm⁻¹

ω and phi scans

Absorption correction: multi-scan

(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.713$, $T_{\max} = 0.747$

13899 measured reflections

2602 independent reflections

2499 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 33.1^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -7 \rightarrow 9$

$k = -14 \rightarrow 12$

$l = -15 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.050$

$S = 1.11$

2602 reflections

111 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0228P)^2 + 0.0624P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.21$ e Å⁻³

$\Delta\rho_{\min} = -0.14$ e Å⁻³

Extinction correction: SHELXL-2018/3

(Sheldrick 2015),

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.034 (6)

Absolute structure: Refined as an inversion twin

Absolute structure parameter: 0.29 (5)

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. Refined as a 2-component inversion twin.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.23376 (4)	0.42563 (3)	0.59746 (2)	0.02230 (7)
N1	0.57372 (15)	0.53197 (10)	0.43072 (8)	0.02210 (18)
H1	0.481 (3)	0.497 (2)	0.4861 (17)	0.046 (5)*
N2	0.70984 (13)	0.65758 (9)	0.29255 (7)	0.01820 (16)
C1	0.75982 (19)	0.46689 (11)	0.39763 (10)	0.0272 (2)
H1A	0.814 (3)	0.3832 (18)	0.4356 (15)	0.034 (4)*
C2	0.84515 (17)	0.54576 (12)	0.31046 (10)	0.0244 (2)
H2	0.975 (3)	0.5379 (16)	0.2678 (15)	0.030 (4)*
C3	0.54531 (15)	0.64734 (11)	0.36651 (9)	0.01807 (17)
C4	0.73381 (19)	0.76456 (11)	0.20131 (9)	0.0250 (2)
H4A	0.872 (3)	0.7478 (19)	0.1651 (14)	0.030 (4)*
H4B	0.715 (3)	0.8557 (16)	0.2354 (13)	0.029 (4)*
H4C	0.619 (3)	0.753 (2)	0.1407 (17)	0.043 (5)*
C5	0.36645 (18)	0.74662 (14)	0.37712 (11)	0.0272 (2)
H5A	0.296 (3)	0.7479 (19)	0.3043 (18)	0.049 (5)*
H5B	0.418 (3)	0.834 (2)	0.3930 (19)	0.063 (6)*
H5C	0.271 (3)	0.713 (2)	0.4340 (17)	0.054 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02118 (10)	0.02475 (11)	0.02096 (11)	−0.00241 (8)	0.00199 (8)	0.00210 (8)
N1	0.0232 (4)	0.0225 (4)	0.0205 (4)	−0.0032 (3)	0.0033 (3)	0.0009 (3)
N2	0.0171 (4)	0.0185 (3)	0.0191 (3)	−0.0025 (3)	0.0016 (3)	−0.0015 (3)
C1	0.0284 (5)	0.0239 (4)	0.0294 (5)	0.0040 (4)	0.0037 (5)	0.0045 (4)
C2	0.0204 (4)	0.0250 (5)	0.0277 (5)	0.0025 (4)	0.0038 (4)	0.0007 (4)
C3	0.0170 (4)	0.0199 (4)	0.0173 (4)	−0.0033 (3)	0.0009 (3)	−0.0035 (3)
C4	0.0286 (5)	0.0219 (4)	0.0245 (4)	−0.0042 (4)	0.0042 (4)	0.0045 (4)
C5	0.0228 (4)	0.0294 (5)	0.0295 (6)	0.0044 (4)	0.0033 (4)	−0.0041 (4)

Geometric parameters (\AA , $^\circ$)

N1—H1	0.923 (19)	C2—H2	0.955 (17)
N1—C1	1.3806 (14)	C3—C5	1.4786 (15)
N1—C3	1.3346 (14)	C4—H4A	0.980 (17)
N2—C2	1.3821 (14)	C4—H4B	0.960 (15)
N2—C3	1.3405 (12)	C4—H4C	1.005 (19)
N2—C4	1.4654 (13)	C5—H5A	0.94 (2)
C1—H1A	0.971 (17)	C5—H5B	0.92 (2)
C1—C2	1.3578 (16)	C5—H5C	0.94 (2)
C1—N1—H1	124.4 (11)	N2—C3—C5	126.54 (10)
C3—N1—H1	125.8 (11)	N2—C4—H4A	106.1 (10)

C3—N1—C1	109.63 (9)	N2—C4—H4B	109.4 (9)
C2—N2—C4	125.43 (9)	N2—C4—H4C	109.6 (11)
C3—N2—C2	109.20 (9)	H4A—C4—H4B	115.3 (15)
C3—N2—C4	125.23 (9)	H4A—C4—H4C	109.4 (13)
N1—C1—H1A	123.3 (10)	H4B—C4—H4C	107.0 (15)
C2—C1—N1	106.69 (10)	C3—C5—H5A	107.2 (12)
C2—C1—H1A	129.9 (10)	C3—C5—H5B	109.3 (14)
N2—C2—H2	121.0 (10)	C3—C5—H5C	108.9 (12)
C1—C2—N2	106.96 (10)	H5A—C5—H5B	109.3 (18)
C1—C2—H2	131.9 (10)	H5A—C5—H5C	108.1 (16)
N1—C3—N2	107.52 (9)	H5B—C5—H5C	113.8 (18)
N1—C3—C5	125.94 (10)		
<hr/>			
N1—C1—C2—N2	0.25 (13)	C3—N1—C1—C2	-0.20 (13)
C1—N1—C3—N2	0.06 (12)	C3—N2—C2—C1	-0.22 (12)
C1—N1—C3—C5	-178.91 (10)	C4—N2—C2—C1	-175.97 (10)
C2—N2—C3—N1	0.10 (11)	C4—N2—C3—N1	175.86 (9)
C2—N2—C3—C5	179.06 (10)	C4—N2—C3—C5	-5.17 (16)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...C11	0.923 (19)	2.122 (19)	3.0396 (10)	172.3 (17)
C2—H2...C11 ⁱ	0.955 (17)	2.695 (17)	3.6084 (11)	160.2 (13)
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