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 3-(1*H*-Tetrazol-5-yl)pyridinium chloride

Jing Dai* and Miao-Jia Yu

 Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
 Correspondence e-mail: fudavid88@yahoo.com.cn

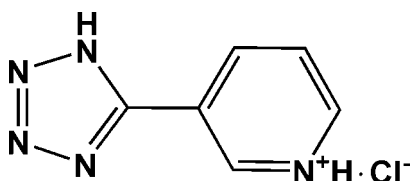
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.104; data-to-parameter ratio = 17.1.

In the cation of the title compound, $\text{C}_6\text{H}_6\text{N}_5^+\cdot\text{Cl}^-$, the pyridinium and tetrazole rings are nearly coplanar, making a dihedral angle of 5.05 (12°). The cations and anions are connected by intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming a centrosymmetric $[2 + 2]$ aggregate. The aggregates are stacked along the a axis.

Related literature

For applications of tetrazole derivatives in coordination chemistry, see: Xiong *et al.* (2002); Wang *et al.* (2005). For the crystal structures of related compounds, see: Dai & Fu (2008); Wen (2008).



Experimental

Crystal data

 $\text{C}_6\text{H}_6\text{N}_5^+\cdot\text{Cl}^-$
 $M_r = 183.61$

 Monoclinic, $P2_1/c$
 $a = 4.2741$ (9) Å

 $b = 8.1992$ (16) Å
 $c = 23.559$ (5) Å
 $\beta = 94.72$ (3) $^\circ$
 $V = 822.8$ (3) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.41$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

 Rigaku Mercury2 diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.883$, $T_{\max} = 0.921$

 8164 measured reflections
 1862 independent reflections
 1431 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.104$
 $S = 1.03$
 1862 reflections

 109 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl1}^{\text{ii}}$	0.86	2.25	3.0625 (18)	157
$\text{N2}-\text{H2}\cdots\text{Cl1}^{\text{ii}}$	0.86	2.23	3.0790 (18)	171

 Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x + 1, y, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: IS2410).

References

- Dai, W. & Fu, D.-W. (2008). *Acta Cryst.* **E64**, o1444.
 Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Wang, X.-S., Tang, Y.-Z., Huang, X.-F., Qu, Z.-R., Che, C.-M., Chan, C. W. H. & Xiong, R.-G. (2005). *Inorg. Chem.* **44**, 5278–5285.
 Wen, X.-C. (2008). *Acta Cryst.* **E64**, m768.
 Xiong, R.-G., Xue, X., Zhao, H., You, X.-Z., Abrahams, B. F. & Xue, Z.-L. (2002). *Angew. Chem. Int. Ed.* **41**, 3800–3803.

supporting information

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3-(1*H*-Tetrazol-5-yl)pyridinium chloride

Jing Dai and Miao-Jia Yu

S1. Comment

In the past few years, more and more people have focused on the chemistry of tetrazole derivatives because of their multiple coordination modes as ligands to metal ions and for the construction of novel metal-organic frameworks (Wang *et al.*, 2005; Xiong *et al.*, 2002; Wen, 2008). We report here the crystal structure of the title compound, 3-(1*H*-tetrazol-5-yl)pyridinium chloride.

In the title compound, the pyridine N atom is protonated (Fig.1). The pyridinium and the tetrazole rings are nearly coplanar and only twisted from each other by a dihedral angle of 5.05 (12) °. The geometric parameters of the tetrazole rings are comparable to those in related molecules (Wang *et al.*, 2005; Dai & Fu, 2008).

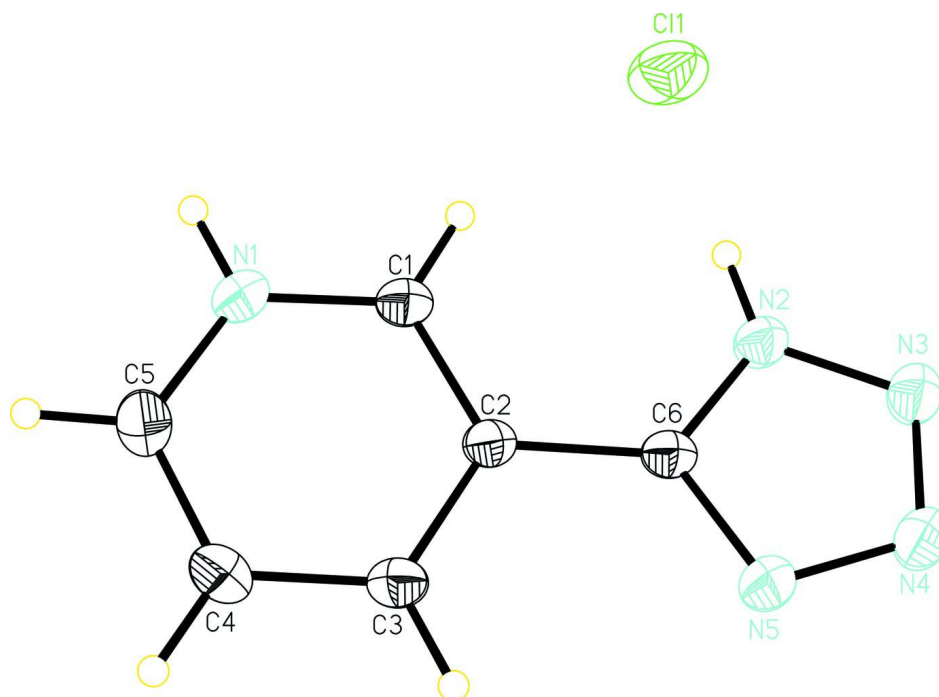
The crystal packing is stabilized by aromatic π - π interactions between the benzene rings of the neighbouring cation systems. The $C_g \cdots C_g^{\text{iii}}$ distance is 4.274 (2) Å; C_g is the centroid of the C1—C6 benzene ring [symmetry code: (iii) $x - 1, y, z$]. The molecular packing is further stabilized by intermolecular N—H \cdots Cl hydrogen bonds (Fig. 2 and Table 1).

S2. Experimental

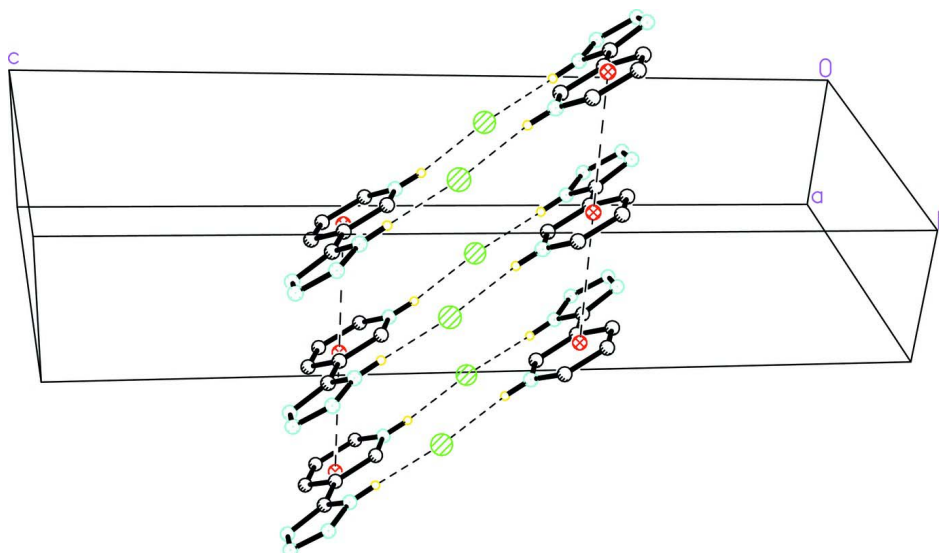
Picolonitrile (30 mmol), NaN₃ (45 mmol), NH₄Cl (33 mmol) and DMF (50 ml) were added in a flask under nitrogen atmosphere and the mixture stirred at 110°C for 20 h. The resulting solution was then poured into ice-water (100 ml), and a white solid was obtained after adding HCl (6 *M*) till pH=6. The precipitate was filtered and washed with distilled water. Colourless block-shaped crystals suitable for X-ray analysis were obtained from the crude product by slow evaporation of an ethanol/HCl (50:1 *v/v*) solution.

S3. Refinement

All H atoms attached to C and N atoms were fixed geometrically and treated as riding, with C—H = 0.93 Å (aromatic) and N—H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C or N})$.

**Figure 1**

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

**Figure 2**

The crystal packing of the title compound, viewed approximately along the *b* axis showing the π - π and N—H...Cl interactions (dotted line) in the title compound. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

3-(1*H*-Tetrazol-5-yl)pyridinium chloride

Crystal data

C₆H₆N₅⁺·Cl⁻ $M_r = 183.61$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 4.2741$ (9) Å $b = 8.1992$ (16) Å $c = 23.559$ (5) Å $\beta = 94.72$ (3)° $V = 822.8$ (3) Å³ $Z = 4$ $F(000) = 376$ $D_x = 1.482$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1862 reflections

 $\theta = 3.0$ – 27.3 ° $\mu = 0.41$ mm⁻¹ $T = 298$ K

Block, colorless

 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Rigaku Mercury2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2005)

 $T_{\min} = 0.883$, $T_{\max} = 0.921$

8164 measured reflections

1862 independent reflections

1431 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.041$ $\theta_{\max} = 27.3$ °, $\theta_{\min} = 3.0$ ° $h = -5 \rightarrow 5$ $k = -10 \rightarrow 10$ $l = -30 \rightarrow 30$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.104$ $S = 1.03$

1862 reflections

109 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-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.2136P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.22$ e Å⁻³ $\Delta\rho_{\min} = -0.28$ 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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.08068 (11)	0.24606 (6)	0.463087 (19)	0.04954 (18)
N1	0.4537 (4)	0.72566 (17)	0.43139 (6)	0.0410 (4)
H1A	0.5437	0.7521	0.4641	0.049*
N2	0.6191 (4)	0.25335 (16)	0.35565 (6)	0.0389 (4)

H2	0.7389	0.2624	0.3867	0.047*
N3	0.5985 (4)	0.12105 (19)	0.32164 (7)	0.0467 (4)
N4	0.3940 (4)	0.1567 (2)	0.27942 (7)	0.0495 (4)
N5	0.2793 (4)	0.31072 (19)	0.28532 (7)	0.0446 (4)
C1	0.5092 (4)	0.5771 (2)	0.41068 (7)	0.0365 (4)
H1	0.6408	0.5050	0.4317	0.044*
C2	0.3697 (4)	0.53090 (19)	0.35769 (6)	0.0307 (4)
C3	0.1717 (4)	0.6434 (2)	0.32759 (7)	0.0373 (4)
H3	0.0751	0.6155	0.2921	0.045*
C4	0.1194 (5)	0.7958 (2)	0.35038 (8)	0.0444 (5)
H4	-0.0111	0.8704	0.3304	0.053*
C5	0.2639 (5)	0.8356 (2)	0.40346 (8)	0.0465 (5)
H5	0.2301	0.9370	0.4196	0.056*
C6	0.4224 (4)	0.3690 (2)	0.33340 (7)	0.0322 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0494 (3)	0.0611 (3)	0.0360 (3)	0.0092 (2)	-0.0094 (2)	-0.0010 (2)
N1	0.0493 (9)	0.0431 (9)	0.0296 (8)	0.0010 (7)	-0.0028 (7)	-0.0053 (6)
N2	0.0425 (8)	0.0389 (8)	0.0337 (8)	0.0066 (6)	-0.0069 (6)	-0.0040 (6)
N3	0.0535 (9)	0.0397 (9)	0.0459 (9)	0.0048 (7)	-0.0019 (7)	-0.0063 (7)
N4	0.0576 (10)	0.0420 (9)	0.0468 (9)	0.0019 (7)	-0.0084 (8)	-0.0098 (7)
N5	0.0528 (9)	0.0392 (8)	0.0391 (8)	0.0018 (7)	-0.0121 (7)	-0.0045 (7)
C1	0.0395 (9)	0.0390 (9)	0.0298 (8)	0.0035 (7)	-0.0042 (7)	0.0021 (7)
C2	0.0321 (8)	0.0331 (8)	0.0265 (8)	-0.0003 (7)	0.0003 (6)	0.0011 (6)
C3	0.0392 (9)	0.0402 (9)	0.0311 (8)	0.0015 (7)	-0.0048 (7)	0.0028 (7)
C4	0.0473 (11)	0.0400 (10)	0.0450 (11)	0.0090 (8)	-0.0014 (9)	0.0073 (8)
C5	0.0560 (12)	0.0356 (10)	0.0482 (11)	0.0051 (9)	0.0066 (9)	-0.0025 (9)
C6	0.0319 (8)	0.0355 (9)	0.0286 (8)	0.0000 (7)	-0.0009 (6)	0.0030 (7)

Geometric parameters (Å, °)

N1—C1	1.340 (2)	C1—C2	1.391 (2)
N1—C5	1.348 (2)	C1—H1	0.9300
N1—H1A	0.8600	C2—C3	1.404 (2)
N2—C6	1.345 (2)	C2—C6	1.470 (2)
N2—N3	1.347 (2)	C3—C4	1.385 (3)
N2—H2	0.8600	C3—H3	0.9300
N3—N4	1.302 (2)	C4—C5	1.387 (3)
N4—N5	1.366 (2)	C4—H4	0.9300
N5—C6	1.331 (2)	C5—H5	0.9300
C1—N1—C5	123.19 (15)	C2—C3—H3	119.8
C1—N1—H1A	118.4	N4—N3—N2	106.29 (14)
C5—N1—H1A	118.4	C3—C4—C5	119.18 (16)
N1—C1—C2	119.91 (15)	C3—C4—H4	120.4
N1—C1—H1	120.0	C5—C4—H4	120.4

C2—C1—H1	120.0	N3—N4—N5	110.72 (14)
C1—C2—C3	118.05 (16)	C6—N5—N4	105.96 (14)
C1—C2—C6	121.81 (14)	N1—C5—C4	119.22 (17)
C3—C2—C6	120.13 (14)	N1—C5—H5	120.4
C6—N2—N3	109.14 (14)	C4—C5—H5	120.4
C6—N2—H2	125.4	N5—C6—N2	107.90 (15)
N3—N2—H2	125.4	N5—C6—C2	125.52 (15)
C4—C3—C2	120.44 (16)	N2—C6—C2	126.58 (14)
C4—C3—H3	119.8		

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 <i>A</i> ...C11 ⁱ	0.86	2.25	3.0625 (18)	157
N2—H2...C11 ⁱⁱ	0.86	2.23	3.0790 (18)	171

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y, z$.