Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2*H*-Tetrazol-5-yl)pyridinium perchlorate monohydrate

Jing Dai* and Wen-Ni Zheng

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

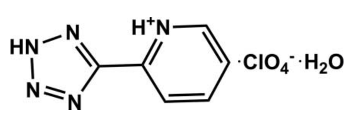
Received 30 November 2009; accepted 3 December 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.065; wR factor = 0.195; data-to-parameter ratio = 17.3.

In the cation of the title compound, $C_6H_6N_5^+\cdot ClO_4^-\cdot H_2O$, the pyridinium and tetrazole rings are essentially coplanar, making a dihedral angle of 1.2 (2)°. In the crystal, intermolecular N—H···O and O—H···O hydrogen bonds link the cations, anions and water molecules into a ribbon-like structure along the c axis. Adjacent ribbons are linked via π–π stacking interactions between the tetrazole rings, with a centroid–centroid distance of 3.484 (2) Å.

Related literature

For applications of tetrazole derivatives in coordination chemistry, see: Zhao *et al.* (2008); Fu *et al.* (2008, 2009). For related structures, see: Fu *et al.* (2007); Fu & Xiong (2008).



Experimental

Crystal data

$C_6H_6N_5^+\cdot ClO_4^-\cdot H_2O$
M_r = 265.62
Triclinic, $P\overline{1}$
a = 7.9945 (16) Å
b = 8.8679 (18) Å
c = 9.4184 (19) Å
α = 78.28 (3)°
β = 70.20 (3)°
γ = 67.97 (3)°
V = 580.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 298 K
0.40 × 0.35 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.881, T_{max} = 0.940
6033 measured reflections
2661 independent reflections
2081 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.195
S = 1.04
2661 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4	0.90	1.76	2.640 (4)	166
N1—H1···O1W^{i}	0.86	1.79	2.633 (4)	166
O1W—H1WB···O3	0.72	2.06	2.778 (4)	172
O1W—H1WA···O2^{ii}	0.78	1.99	2.771 (4)	174

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+1, -y, -z+2$.

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*.

This work was supported by a start-up grant from Southeast University to Professor Ren-Gen Xiong.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2980).

References

Fu, D.-W., Ge, J.-Z., Dai, J., Ye, H.-Y. & Qu, Z.-R. (2009). *Inorg. Chem. Commun.* **12**, 994–997.
Fu, D.-W., Song, Y.-M., Wang, G.-X., Ye, Q., Xiong, R.-G., Akutagawa, T., Nakamura, T., Chan, P. W. H. & Huang, S.-D. (2007). *J. Am. Chem. Soc.* **129**, 5346–5347.
Fu, D.-W. & Xiong, R.-G. (2008). *Dalton Trans.* pp. 3946–3948.
Fu, D.-W., Zhang, W. & Xiong, R.-G. (2008). *Cryst. Growth Des.* **8**, 3461–3464.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhao, H., Qu, Z.-R., Ye, H.-Y. & Xiong, R.-G. (2008). *Chem. Soc. Rev.* **37**, 84–100.

supplementary materials

Acta Cryst. (2010). E**66**, o71 [**doi:10.1107/S160053680905199X**]

2-(2*H*-Tetrazol-5-yl)pyridinium perchlorate monohydrate

J. Dai and **W.-N. Zheng**

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 (Zhao *et al.*, 2008; Fu *et al.*, 2008). As an extension of these work on the structure and properties (Fu *et al.*, 2007; Fu & Xiong 2008), we report here the crystal structure of the title compound 2-(2*H*-tetrazol-5-yl)pyridinium perchlorate monohydrate.

In the title compound (Fig.1), the pyridine N atom is protonated. The pyridinium and tetrazole rings are essentially co-planar, with the dihedral angle between them being 1.2 (2)°. The geometric parameters of the tetrazole rings are comparable to those in related structures (Zhao *et al.*, 2008; Fu *et al.*, 2009).

The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds. These hydrogen bonds link the ionic units and water molecules to form a ribbon like structure parallel to the *c* axis (Table 1 and Fig.2).

Experimental

Picolinonitrile (30 mmol), NaN_3 (45 mmol), NH_4Cl (33 mmol) and DMF (50 ml) were added in a flask under nitrogen atmosphere and the mixture was 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 a water-$HClO_4$ (50:1 *v*/*v*) solution.

Refinement

All H atoms attached to C and N atoms were fixed geometrically and treated as riding with C–H = 0.93 Å (aromatic), N–H = 0.86 Å (pyridine N) and N–H = 0.90 Å (tetrazole N) with U_{iso}(H) =1.2U_{eq}(C,N). H atoms of the water molecule were located in difference Fourier maps and freely refined. In the last stage of the refinement they were treated as riding on the O atom, with U_{iso}(H) = 1.5U_{eq}(O).

Figures

Fig. 1. The asymmetric unit of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. The crystal packing of the title compound, viewed along the *a* axis, showing the two dimensionnal hydrogen-bonded network. H atoms not involved in hydrogen bonding (dashed line) have been omitted for clarity.

2-(2H-Tetrazol-5-yl)pyridinium perchlorate monohydrate

Crystal data

$C_6H_6N_5^+ \cdot ClO_4^- \cdot H_2O$	$Z = 2$
$M_r = 265.62$	$F(000) = 272$
Triclinic, $P\overline{1}$	$D_x = 1.521$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.9945$ (16) Å	Cell parameters from 2081 reflections
$b = 8.8679$ (18) Å	$\theta = 3.1–27.5°$
$c = 9.4184$ (19) Å	$\mu = 0.35$ mm^{-1}
$\alpha = 78.28$ (3)°	$T = 298$ K
$\beta = 70.20$ (3)°	Block, colourless
$\gamma = 67.97$ (3)°	0.40 × 0.35 × 0.20 mm
$V = 580.1$ (2) Å^3	

Data collection

Rigaku Mercury2 diffractometer	2661 independent reflections
Radiation source: fine-focus sealed tube	2081 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.026$
Detector resolution: 13.6612 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
ω scans	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$k = -11 \rightarrow 11$
$T_{min} = 0.881$, $T_{max} = 0.940$	$l = -12 \rightarrow 12$
6033 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.065$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.195$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0988P)^2 + 0.7631P]$ where $P = (F_o^2 + 2F_c^2)/3$
2661 reflections	$(\Delta/\sigma)_{max} = 0.001$
154 parameters	$\Delta\rho_{max} = 0.53$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.61$ 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)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2411 (4)	0.5225 (3)	0.0449 (3)	0.0337 (6)
H1	0.3305	0.4346	0.0590	0.040*
N2	0.3244 (4)	0.4372 (3)	0.3287 (3)	0.0398 (7)
N3	0.3065 (4)	0.4464 (3)	0.4707 (3)	0.0411 (7)
H3	0.3806	0.3754	0.5256	0.049*
N4	0.1668 (5)	0.5728 (4)	0.5322 (3)	0.0459 (7)
N5	0.0848 (4)	0.6535 (3)	0.4264 (3)	0.0412 (7)
C1	0.2097 (6)	0.5603 (5)	−0.0901 (4)	0.0445 (8)
H1A	0.2857	0.4927	−0.1676	0.053*
C2	0.0668 (6)	0.6976 (5)	−0.1161 (4)	0.0525 (10)
H2A	0.0448	0.7239	−0.2104	0.063*
C3	−0.0443 (6)	0.7964 (5)	0.0006 (4)	0.0517 (10)
H3A	−0.1431	0.8896	−0.0144	0.062*
C4	−0.0087 (5)	0.7568 (4)	0.1392 (4)	0.0442 (8)
H4A	−0.0825	0.8233	0.2178	0.053*
C5	0.1372 (4)	0.6176 (4)	0.1602 (3)	0.0330 (7)
C6	0.1842 (4)	0.5680 (4)	0.3032 (3)	0.0323 (7)
Cl1	0.58684 (12)	0.09352 (10)	0.68911 (9)	0.0393 (3)
O1	0.7478 (6)	0.0326 (6)	0.5118 (5)	0.0995 (13)
O2	0.4562 (4)	0.0041 (4)	0.7313 (3)	0.0565 (7)
O3	0.6963 (4)	0.0591 (3)	0.7938 (3)	0.0557 (8)
O4	0.4923 (4)	0.2690 (3)	0.6656 (3)	0.0501 (7)
O1W	0.5256 (4)	0.2475 (3)	1.0393 (3)	0.0483 (7)
H1WA	0.5261	0.1815	1.1080	0.073*
H1WB	0.5728	0.2054	0.9710	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0345 (14)	0.0324 (13)	0.0324 (13)	−0.0090 (11)	−0.0084 (11)	−0.0057 (10)
N2	0.0450 (16)	0.0349 (14)	0.0343 (14)	−0.0017 (12)	−0.0165 (12)	−0.0060 (11)
N3	0.0482 (16)	0.0363 (15)	0.0360 (14)	−0.0055 (13)	−0.0197 (13)	−0.0010 (11)

N4	0.0537 (18)	0.0441 (16)	0.0345 (15)	−0.0054 (14)	−0.0158 (13)	−0.0079 (12)
N5	0.0465 (16)	0.0368 (15)	0.0326 (14)	−0.0002 (12)	−0.0140 (12)	−0.0088 (11)
C1	0.055 (2)	0.049 (2)	0.0323 (17)	−0.0221 (18)	−0.0079 (15)	−0.0086 (14)
C2	0.062 (2)	0.062 (2)	0.0369 (18)	−0.019 (2)	−0.0240 (17)	0.0028 (17)
C3	0.051 (2)	0.049 (2)	0.048 (2)	−0.0013 (17)	−0.0260 (18)	0.0017 (17)
C4	0.0438 (19)	0.0415 (19)	0.0392 (18)	−0.0013 (15)	−0.0151 (15)	−0.0058 (14)
C5	0.0333 (15)	0.0332 (15)	0.0315 (15)	−0.0083 (13)	−0.0110 (12)	−0.0030 (12)
C6	0.0336 (15)	0.0296 (15)	0.0309 (15)	−0.0059 (12)	−0.0094 (12)	−0.0053 (12)
Cl1	0.0429 (5)	0.0358 (4)	0.0364 (4)	−0.0004 (3)	−0.0210 (3)	−0.0054 (3)
O1	0.086 (3)	0.122 (3)	0.083 (3)	−0.016 (3)	−0.014 (2)	−0.046 (2)
O2	0.0581 (17)	0.0598 (17)	0.0539 (16)	−0.0220 (14)	−0.0222 (13)	0.0051 (13)
O3	0.0647 (17)	0.0527 (16)	0.0510 (15)	0.0011 (13)	−0.0408 (14)	−0.0056 (12)
O4	0.0583 (16)	0.0351 (13)	0.0477 (14)	0.0082 (11)	−0.0281 (12)	−0.0083 (11)
O1W	0.0575 (16)	0.0363 (13)	0.0405 (13)	−0.0069 (12)	−0.0088 (11)	−0.0067 (10)

Geometric parameters (Å, °)

N1—C1	1.331 (4)	C2—H2A	0.93
N1—C5	1.348 (4)	C3—C4	1.378 (5)
N1—H1	0.86	C3—H3A	0.93
N2—N3	1.312 (4)	C4—C5	1.378 (5)
N2—C6	1.324 (4)	C4—H4A	0.93
N3—N4	1.314 (4)	C5—C6	1.458 (4)
N3—H3	0.90	Cl1—O2	1.446 (3)
N4—N5	1.318 (4)	Cl1—O3	1.447 (3)
N5—C6	1.354 (4)	Cl1—O4	1.460 (3)
C1—C2	1.368 (6)	Cl1—O1	1.768 (4)
C1—H1A	0.93	O1W—H1WA	0.78
C2—C3	1.382 (6)	O1W—H1WB	0.72
C1—N1—C5	122.0 (3)	C2—C3—H3A	120.0
C1—N1—H1	119.0	C3—C4—C5	119.4 (3)
C5—N1—H1	119.0	C3—C4—H4A	120.3
N3—N2—C6	101.6 (3)	C5—C4—H4A	120.3
N2—N3—N4	114.4 (3)	N1—C5—C4	119.2 (3)
N2—N3—H3	125.6	N1—C5—C6	118.2 (3)
N4—N3—H3	120.0	C4—C5—C6	122.6 (3)
N3—N4—N5	106.4 (3)	N2—C6—N5	112.5 (3)
N4—N5—C6	105.1 (3)	N2—C6—C5	125.1 (3)
N1—C1—C2	120.7 (3)	N5—C6—C5	122.3 (3)
N1—C1—H1A	119.7	O2—Cl1—O3	113.55 (17)
C2—C1—H1A	119.7	O2—Cl1—O4	111.66 (17)
C1—C2—C3	118.7 (3)	O3—Cl1—O4	110.80 (16)
C1—C2—H2A	120.7	O2—Cl1—O1	107.3 (2)
C3—C2—H2A	120.7	O3—Cl1—O1	107.05 (19)
C4—C3—C2	120.0 (4)	O4—Cl1—O1	106.0 (2)
C4—C3—H3A	120.0	H1WA—O1W—H1WB	107.6

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O4	0.90	1.76	2.640 (4)	166
N1—H1···O1W^{i}	0.86	1.79	2.633 (4)	166
O1W—H1WB···O3	0.72	2.06	2.778 (4)	172
O1W—H1WA···O2^{ii}	0.78	1.99	2.771 (4)	174

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

Fig. 1

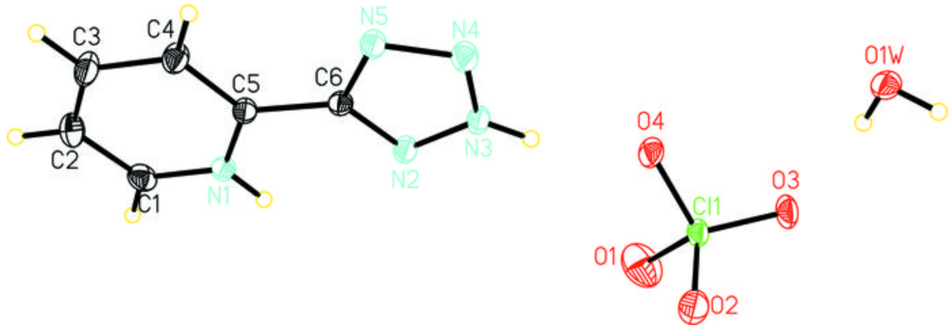


Fig. 2