

3-Hydroxypyridinium perchlorate

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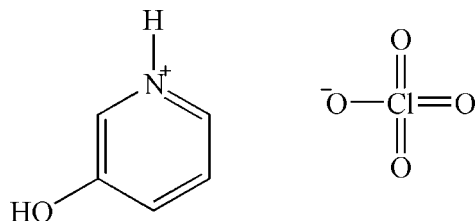
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.032; wR factor = 0.085; data-to-parameter ratio = 12.4.

In the crystal structure of the title compound, $\text{C}_5\text{H}_6\text{NO}^+\cdot\text{ClO}_4^-$, intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds between the cations and anions result in the formation of an infinite two-dimensional hydrogen-bonded supramolecular network.

Related literature

For related literature, see: Breeze & Wang (1993); Castillo *et al.* (2000); Fukunaga *et al.* (2004); Gao *et al.* (2004, 2005); Kawata *et al.* (1997); Szafran *et al.* (1998); Zhao *et al.* (2004).



Experimental

Crystal data

$\text{C}_5\text{H}_6\text{NO}^+\cdot\text{ClO}_4^-$

$M_r = 195.56$

Triclinic, $P\bar{1}$

$a = 5.289$ (5) Å

$b = 8.308$ (8) Å

$c = 9.047$ (8) Å

$\alpha = 100.346$ (9)°

$\beta = 94.286$ (9)°

$\gamma = 101.732$ (9)°

$V = 380.4$ (6) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 0.48$ mm⁻¹

$T = 291$ (2) K

$0.33 \times 0.22 \times 0.20$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.856$, $T_{\max} = 0.909$

2682 measured reflections

1375 independent reflections

1266 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.085$

$S = 1.04$

1375 reflections

111 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.29$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{i}}$	0.86	2.34	3.025 (3)	137
$\text{N1}-\text{H1B}\cdots\text{O2}^{\text{ii}}$	0.86	2.32	2.941 (3)	130
$\text{O1}-\text{H1}\cdots\text{O5}^{\text{iii}}$	0.82	2.00	2.818 (3)	174
$\text{C5}-\text{H5}\cdots\text{O4}^{\text{iv}}$	0.93	2.58	3.270 (4)	131

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

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2; data reduction: SAINT (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998) and PLATON (Spek, 2003); 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: PR2015).

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supplementary materials

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3-Hydroxypyridinium perchlorate

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Comment

3-Hydroxypyridine (3-HP) has one hydrogen bond accepting heterocyclic nitrogen and a hydrogen bond donating hydroxy group, which is not only capable of binding to metal centers (Kawata *et al.*, 1997; Castillo *et al.*, 2000; Gao *et al.*, 2005), but can also form cyclic hydrogen-bonding polymers with trifluoroacetic acid and tartronic acid (Breeze & Wang, 1993; Fukunaga *et al.*, 2004). The crystal structures of some hydroxy- substituted zwitterionic pyridinioacetates have been determined to elucidate the interaction of hydrogen bonds (Zhao *et al.*, 2004; Gao *et al.*, 2004; Szafran *et al.*, 1998). Our interest has been directed toward the synthesis of a metal complex based on (3-HP) and 3,5-pyrazoledicarboxylic acid, however, the reaction yielded the title organic compound, (I), whose crystal structure is reported here. Compound (I) consists of one 3-hydroxypyridinium cation and one perchlorate anion (Fig. 1). A two-dimensional supramolecular framework is formed *via* intermolecular N—H \cdots O, O—H \cdots O, and C—H \cdots O hydrogen bonds between the hydroxy group and nitrogen of 3-hydroxypyridinium and oxygen atoms (O2, O3 and O4) of the perchlorate (Table 1 and Fig. 2).

Experimental

All reagents were of AR grade and were used without further purification. An aqueous solution of 3-hydroxypyridine (1.0 mmol), 3,5-pyrazoledicarboxylic acid (0.50 mmol), and sodium hydroxide (4.00 g, 1.0 mmol) was reacted with an aqueous solution of Cu(ClO₄)₂·6H₂O (0.5 mmol) (0.50 mmol). The pH of the mixture was treated with 0.05 M perchloric acid to a pH of 2–3 and was refluxed for 8 h, colorless crystals of the title compound separated from the filtrate after several days, washed with distilled water and dried in air. Analysis calculated for C₅H₆NC₄O₄: C 30.71, H 3.09, N 7.16%; found: C 31.15, H 3.14, N 7.68%.

Refinement

All H atoms attached to C atoms were treated as riding, with C—H = 0.930 Å (aromatic and heteroaromatic) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ of the carrier atoms. The O—H distances were fixed at 0.82 Å, H atoms attached to N atom was constrained in the riding model, with N—H = 0.860 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ of the carrier atoms.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINTE* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker 1998) and *PLATON* (Spek 2003); software used to prepare material for publication: *SHELXTL*.

Figures

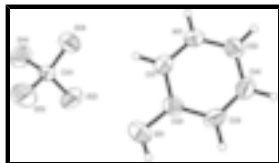


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and 50% displacement ellipsoids.

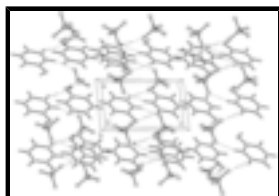


Fig. 2. Packing diagram of compound (I), showing the interactions of N—H \cdots O, O—H \cdots O, and C—H \cdots O hydrogen bonds, shown as dashed lines.

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Crystal data

C₅H₆NO⁺·ClO₄⁻

$M_r = 195.56$

Triclinic, $P\bar{1}$

$a = 5.289$ (5) Å

$b = 8.308$ (8) Å

$c = 9.047$ (8) Å

$\alpha = 100.346$ (9)°

$\beta = 94.286$ (9)°

$\gamma = 101.732$ (9)°

$V = 380.4$ (6) Å³

$Z = 2$

$F_{000} = 200$

$D_x = 1.707$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1662 reflections

$\theta = 2.3$ – 28.2 °

$\mu = 0.48$ mm⁻¹

$T = 291$ (2) K

Block, colourless

$0.33 \times 0.22 \times 0.20$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ (2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.856$, $T_{\max} = 0.909$

2682 measured reflections

1375 independent reflections

1266 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 25.5$ °

$\theta_{\text{min}} = 2.3$ °

$h = -6 \rightarrow 6$

$k = -10 \rightarrow 10$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.1807P]$
$wR(F^2) = 0.085$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} < 0.001$
1375 reflections	$\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
111 parameters	$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.162 (12)

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.99857 (8)	0.81013 (6)	0.26437 (5)	0.0376 (2)
O1	0.8190 (3)	0.5623 (2)	0.61642 (19)	0.0610 (5)
H1	0.8863	0.5079	0.6687	0.091*
O2	1.1783 (3)	0.8632 (2)	0.39855 (18)	0.0700 (5)
O3	0.7448 (3)	0.8283 (3)	0.2972 (2)	0.0661 (5)
O4	1.0803 (3)	0.9099 (2)	0.15520 (18)	0.0625 (5)
O5	0.9893 (4)	0.6374 (2)	0.2005 (2)	0.0739 (5)
N1	0.3914 (3)	0.8349 (2)	0.70012 (19)	0.0437 (4)
H1B	0.3169	0.8974	0.6535	0.052*
C1	0.5440 (4)	0.7477 (3)	0.6254 (2)	0.0427 (5)
H1A	0.5680	0.7548	0.5258	0.051*
C2	0.6662 (4)	0.6466 (2)	0.6984 (2)	0.0398 (4)
C3	0.6274 (4)	0.6413 (3)	0.8472 (2)	0.0467 (5)
H3	0.7100	0.5756	0.8988	0.056*
C4	0.4666 (4)	0.7332 (3)	0.9187 (2)	0.0483 (5)
H4	0.4392	0.7287	1.0183	0.058*
C5	0.3469 (4)	0.8310 (3)	0.8432 (2)	0.0447 (5)
H5	0.2373	0.8933	0.8903	0.054*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0429 (3)	0.0432 (3)	0.0325 (3)	0.0218 (2)	0.00492 (18)	0.0080 (2)
O1	0.0726 (11)	0.0589 (10)	0.0620 (10)	0.0379 (8)	0.0183 (9)	0.0088 (8)
O2	0.0756 (12)	0.0865 (13)	0.0473 (9)	0.0272 (10)	-0.0159 (8)	0.0106 (9)
O3	0.0565 (10)	0.0965 (14)	0.0647 (10)	0.0431 (9)	0.0234 (8)	0.0301 (10)
O4	0.0670 (11)	0.0742 (12)	0.0537 (10)	0.0157 (9)	0.0131 (8)	0.0290 (9)
O5	0.0982 (14)	0.0493 (10)	0.0754 (12)	0.0333 (9)	0.0026 (10)	-0.0007 (9)
N1	0.0464 (9)	0.0484 (10)	0.0432 (9)	0.0209 (8)	0.0028 (7)	0.0161 (8)
C1	0.0488 (11)	0.0472 (12)	0.0354 (10)	0.0149 (9)	0.0051 (8)	0.0114 (9)
C2	0.0421 (10)	0.0344 (10)	0.0425 (10)	0.0114 (8)	0.0031 (8)	0.0039 (8)
C3	0.0562 (12)	0.0436 (11)	0.0460 (11)	0.0180 (9)	0.0005 (9)	0.0175 (9)
C4	0.0601 (13)	0.0523 (12)	0.0358 (10)	0.0156 (10)	0.0087 (9)	0.0120 (9)
C5	0.0445 (11)	0.0457 (11)	0.0453 (11)	0.0154 (9)	0.0079 (9)	0.0057 (9)

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

C11—O2	1.4272 (19)	C1—C2	1.384 (3)
C11—O3	1.428 (2)	C1—H1A	0.9300
C11—O4	1.4361 (18)	C2—C3	1.384 (3)
C11—O5	1.437 (2)	C3—C4	1.376 (3)
O1—C2	1.352 (2)	C3—H3	0.9300
O1—H1	0.8200	C4—C5	1.368 (3)
N1—C1	1.333 (3)	C4—H4	0.9300
N1—C5	1.338 (3)	C5—H5	0.9300
N1—H1B	0.8600		
O2—C11—O3	110.49 (12)	O1—C2—C1	115.95 (19)
O2—C11—O4	109.76 (12)	O1—C2—C3	125.39 (18)
O3—C11—O4	108.84 (10)	C1—C2—C3	118.65 (19)
O2—C11—O5	109.78 (12)	C4—C3—C2	119.95 (18)
O3—C11—O5	109.27 (13)	C4—C3—H3	120.0
O4—C11—O5	108.66 (13)	C2—C3—H3	120.0
C2—O1—H1	109.5	C5—C4—C3	120.06 (19)
C1—N1—C5	123.99 (17)	C5—C4—H4	120.0
C1—N1—H1B	118.0	C3—C4—H4	120.0
C5—N1—H1B	118.0	N1—C5—C4	118.38 (19)
N1—C1—C2	118.96 (19)	N1—C5—H5	120.8
N1—C1—H1A	120.5	C4—C5—H5	120.8
C2—C1—H1A	120.5		
C5—N1—C1—C2	-0.2 (3)	C1—C2—C3—C4	1.1 (3)
N1—C1—C2—O1	-179.72 (18)	C2—C3—C4—C5	-0.6 (3)
N1—C1—C2—C3	-0.7 (3)	C1—N1—C5—C4	0.6 (3)
O1—C2—C3—C4	180.0 (2)	C3—C4—C5—N1	-0.2 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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N1—H1B···O3 ⁱ	0.86	2.34	3.025 (3)	137
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O1—H1···O5 ⁱⁱⁱ	0.82	2.00	2.818 (3)	174
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Fig. 1

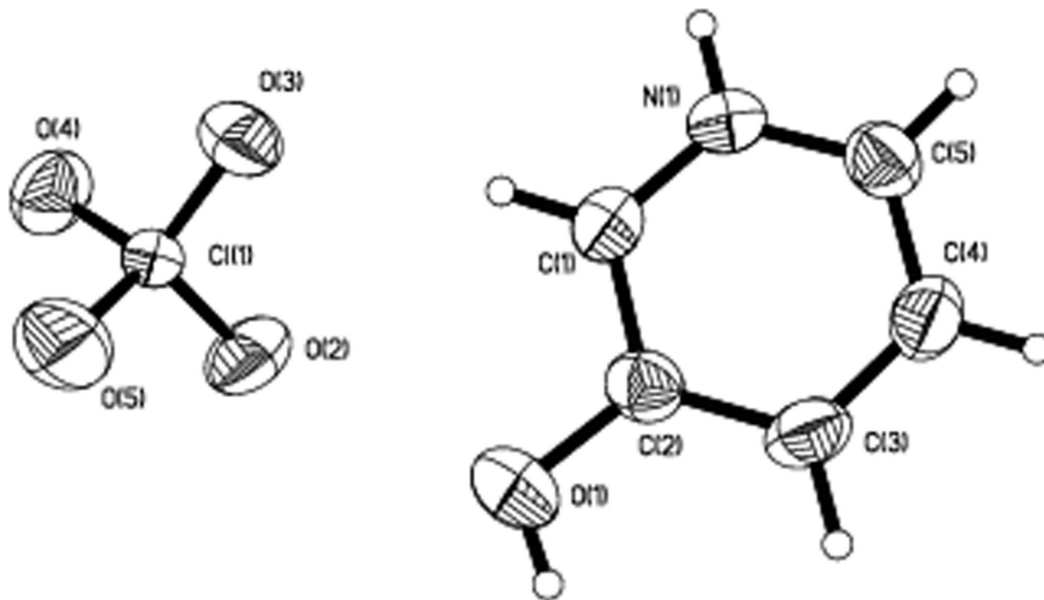


Fig. 2

