Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Hydroxypyridinium hydrogen sulfate

Li-Hua Huo,[a] Ying-Ming Xu,[a] Shan Gao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

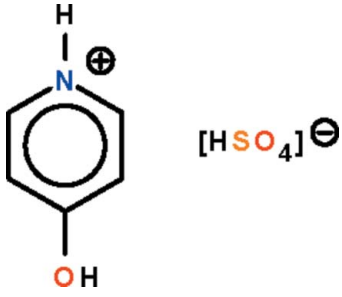
Received 14 November 2009; accepted 15 November 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 12.6.

The crystal structure of the title salt, $C_5H_6NO^+\cdot HSO_4^-$, consists of planar(r.m.s. deviation = 0.001 Å) 4-hydroxypyridinium cations and hydrogen sulfate anions which are hydrogen bonded into a layer motif. In the anion, the S—O bond [1.551 (2) Å] involving the O atom bearing the acid H atom is longer than the other three S—O bonds, which range from 1.437 (1) to 1.454 (1) Å.

Related literature

For the crystal structures of bis(4-hydroxypyridinium) sulfate monohydrate and tris(4-hydroxypyridinium) hydrogen disulfate monohydrate, see: Xu *et al.* (2009*a*,*b*).



Experimental

Crystal data

$C_5H_6NO^+\cdot HSO_4^-$
M_r = 193.18
Monoclinic, $P2_1/c$
a = 10.4541 (7) Å
b = 10.7017 (6) Å
c = 6.8397 (4) Å
β = 96.503 (2)°
V = 760.28 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 293 K
0.27 × 0.21 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.898, T_{max} = 0.941
7273 measured reflections
1729 independent reflections
1612 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.03
1729 reflections
137 parameters
7 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—O1	1.445 (1)	S1—O3	1.437 (1)
S1—O2	1.551 (2)	S1—O4	1.454 (1)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O4^{i}	0.85 (1)	1.77 (1)	2.603 (2)	168 (3)
O5—H5···O1	0.85 (1)	1.77 (1)	2.6166 (19)	175 (3)
N1—H1···O3^{ii}	0.84 (1)	2.04 (1)	2.8529 (19)	163 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the Key Project of the Natural Science Foundation of Heilongjiang Province (No. ZD200903), the Scientific Fund of Remarkable Teachers of Heilongjiang Province (No. 1054 G036), Heilongjiang University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2677).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.
Xu, Y.-M., Gao, S. & Ng, S. W. (2009*a*). *Acta Cryst.* E**65**, o3146.
Xu, Y.-M., Gao, S. & Ng, S. W. (2009*b*). *Acta Cryst.* E**65**, o3147.

supporting information

Acta Cryst. (2009). E**65**, o3148 [doi:10.1107/S1600536809048533]

4-Hydroxypyridinium hydrogen sulfate

Li-Hua Huo, Ying-Ming Xu, Shan Gao and Seik Weng Ng

S1. Experimental

The compound is a side product that was obtained when commercially available 4-hydroxypyridine-3-sulfonic acid was recrystallized from water. Its crystals were obtained from a water solution.

S2. Refinement

Carbon-bound H-atoms refined with a C–H distance restraint of 0.95±0.01 Å; their temperature factors were refined. The nitrogen- and oxygen-bound H-atoms were refined with a distance restraint of N–H = O–H = 0.85±0.01 Å; their temperature factors were refined.

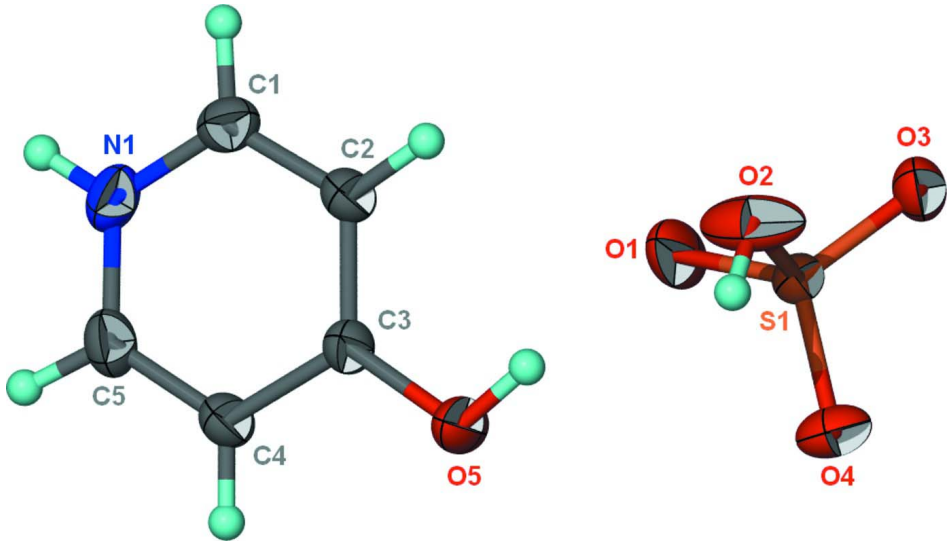


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[C_5H_6NO]^+$ $[HSO_4]^-$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-Hydroxypyridinium hydrogen sulfate

Crystal data

$C_5H_6NO^+ \cdot HSO_4^-$
M_r = 193.18
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.4541 (7) Å
b = 10.7017 (6) Å
c = 6.8397 (4) Å
β = 96.503 (2)°
V = 760.28 (8) Å^3
Z = 4
F(000) = 400
D_x = 1.688 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6627 reflections

θ = 3.6–27.4°
μ = 0.41 mm^{-1}
T = 293 K

Prism, colorless
0.27 × 0.21 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.898, T_{max} = 0.941

7273 measured reflections
1729 independent reflections
1612 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 27.4°, θ_{min} = 3.6°
h = −13→13
k = −13→13
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.098
S = 1.03
1729 reflections
137 parameters
7 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.3712P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.15352 (4)	0.36664 (3)	0.22053 (6)	0.02984 (15)
O1	0.28767 (13)	0.37320 (14)	0.2986 (3)	0.0580 (4)
O2	0.1438 (2)	0.41518 (14)	0.0056 (2)	0.0657 (5)
O3	0.07735 (11)	0.45271 (12)	0.3196 (2)	0.0423 (3)
O4	0.10415 (14)	0.23950 (12)	0.21136 (18)	0.0430 (3)
O5	0.46894 (12)	0.20467 (13)	0.2977 (2)	0.0486 (4)
N1	0.82340 (14)	0.36324 (15)	0.3509 (2)	0.0416 (4)
C1	0.72047 (18)	0.43863 (17)	0.3364 (3)	0.0388 (4)
C2	0.59869 (16)	0.39037 (16)	0.3180 (3)	0.0351 (4)
C3	0.58308 (15)	0.26042 (15)	0.3148 (2)	0.0320 (3)
C4	0.69235 (16)	0.18433 (16)	0.3312 (3)	0.0359 (4)
C5	0.81115 (16)	0.23848 (19)	0.3484 (3)	0.0410 (4)
H1	0.8975 (14)	0.395 (2)	0.366 (4)	0.063 (7)*
H2	0.134 (3)	0.357 (2)	−0.078 (4)	0.079 (9)*
H5	0.412 (2)	0.2615 (19)	0.293 (4)	0.063 (7)*
H1A	0.735 (2)	0.5257 (10)	0.338 (3)	0.044 (6)*
H2A	0.5283 (16)	0.4452 (18)	0.302 (3)	0.049 (6)*
H4	0.6824 (19)	0.0967 (9)	0.330 (3)	0.040 (5)*
H5A	0.8881 (16)	0.191 (2)	0.356 (4)	0.061 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0273 (2)	0.0272 (2)	0.0352 (2)	−0.00275 (13)	0.00428 (15)	−0.00138 (13)
O1	0.0264 (7)	0.0495 (9)	0.0963 (12)	0.0041 (5)	−0.0012 (7)	−0.0159 (8)
O2	0.1253 (16)	0.0347 (7)	0.0384 (7)	−0.0135 (9)	0.0154 (8)	0.0017 (6)
O3	0.0312 (6)	0.0428 (7)	0.0538 (8)	0.0005 (5)	0.0090 (5)	−0.0101 (6)
O4	0.0581 (8)	0.0317 (6)	0.0391 (6)	−0.0130 (5)	0.0045 (5)	0.0004 (5)
O5	0.0254 (6)	0.0346 (7)	0.0852 (11)	−0.0021 (5)	0.0041 (6)	−0.0049 (6)
N1	0.0289 (7)	0.0507 (9)	0.0457 (8)	−0.0105 (6)	0.0064 (6)	−0.0084 (7)
C1	0.0439 (9)	0.0337 (8)	0.0397 (9)	−0.0068 (7)	0.0086 (7)	−0.0045 (7)
C2	0.0329 (8)	0.0303 (8)	0.0424 (9)	0.0037 (6)	0.0052 (7)	−0.0022 (6)
C3	0.0268 (7)	0.0316 (8)	0.0376 (8)	−0.0003 (6)	0.0037 (6)	−0.0026 (6)
C4	0.0315 (8)	0.0308 (8)	0.0454 (9)	0.0034 (6)	0.0037 (7)	−0.0031 (7)
C5	0.0279 (8)	0.0485 (10)	0.0466 (9)	0.0043 (7)	0.0040 (7)	−0.0058 (8)

Geometric parameters (Å, º)

S1—O1	1.445 (1)	N1—H1	0.841 (10)
S1—O2	1.551 (2)	C1—C2	1.366 (2)
S1—O3	1.437 (1)	C1—H1A	0.945 (10)
S1—O4	1.454 (1)	C2—C3	1.400 (2)
S1—O2	1.5514 (15)	C2—H2A	0.938 (10)
O2—H2	0.848 (10)	C3—C4	1.397 (2)
O5—C3	1.3273 (19)	C4—C5	1.363 (2)
O5—H5	0.849 (10)	C4—H4	0.943 (9)
N1—C1	1.340 (2)	C5—H5A	0.947 (10)
N1—C5	1.341 (3)		
O3—S1—O1	111.16 (8)	C2—C1—H1A	121.6 (13)
O3—S1—O4	114.04 (8)	C1—C2—C3	118.86 (16)
O1—S1—O4	112.73 (9)	C1—C2—H2A	119.0 (14)
O3—S1—O2	104.60 (9)	C3—C2—H2A	122.1 (14)
O1—S1—O2	106.91 (11)	O5—C3—C4	117.63 (15)
O4—S1—O2	106.71 (8)	O5—C3—C2	123.36 (15)
S1—O2—H2	113 (2)	C4—C3—C2	119.01 (15)
C3—O5—H5	107.5 (19)	C5—C4—C3	119.20 (16)
C1—N1—C5	121.59 (15)	C5—C4—H4	121.5 (12)
C1—N1—H1	119.2 (19)	C3—C4—H4	119.3 (12)
C5—N1—H1	119 (2)	N1—C5—C4	120.59 (16)
N1—C1—C2	120.75 (16)	N1—C5—H5A	116.9 (16)
N1—C1—H1A	117.6 (13)	C4—C5—H5A	122.5 (16)
C5—N1—C1—C2	−0.1 (3)	O5—C3—C4—C5	179.79 (17)
N1—C1—C2—C3	0.1 (3)	C2—C3—C4—C5	−0.4 (3)
C1—C2—C3—O5	179.94 (17)	C1—N1—C5—C4	−0.1 (3)
C1—C2—C3—C4	0.2 (2)	C3—C4—C5—N1	0.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O4^{i}	0.85 (1)	1.77 (1)	2.603 (2)	168 (3)
O5—H5···O1	0.85 (1)	1.77 (1)	2.6166 (19)	175 (3)
N1—H1···O3^{ii}	0.84 (1)	2.04 (1)	2.8529 (19)	163 (2)

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