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4-Hydroxypyridinium hydrogen sulfate

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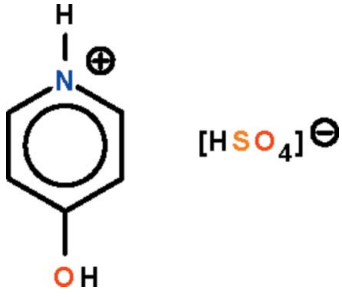
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.098; data-to-parameter ratio = 12.6.

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).

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

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supplementary materials

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4-Hydroxypyridinium hydrogen sulfate

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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.

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.

Figures

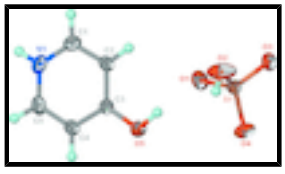

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $[C_5H_6NO]^+$ $[HSO_4]^-$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-Hydroxypyridinium hydrogen sulfate

Crystal data

$C_5H_6N_1O_1^+ \cdot HSO_4^-$	F_{000} = 400
M_r = 193.18	D_x = 1.688 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6627 reflections
a = 10.4541 (7) Å	θ = 3.6–27.4º
b = 10.7017 (6) Å	μ = 0.41 mm^{-1}
c = 6.8397 (4) Å	T = 293 K
β = 96.503 (2)º	Prism, colorless
V = 760.28 (8) Å^3	0.27 × 0.21 × 0.15 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	1729 independent reflections
Radiation source: fine-focus sealed tube	1612 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.017

T = 293 K	θ_{max} = 27.4º
ω scan	θ_{min} = 3.6º
Absorption correction: Multi-scan (ABSCOR; Higashi, 1995)	h = −13→13
T_{min} = 0.898, T_{max} = 0.941	k = −13→13
7273 measured reflections	l = −8→8

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.036	All H-atom parameters refined
$wR(F^2)$ = 0.098	$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.3712P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
1729 reflections	$\Delta\rho_{max}$ = 0.52 e Å^{-3}
137 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
7 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	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, °)

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, °)

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*.

Fig. 1