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4-Hydroxypyridinium-3-sulfonate

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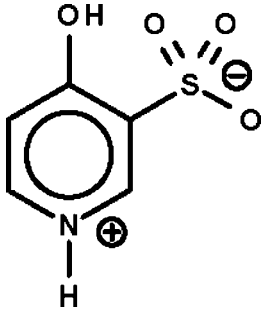
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.068; data-to-parameter ratio = 12.0.

The reaction of 4-hydroxypyridine and oleum produces 4-hydroxypyridinium-3-sulfonate, $C_5H_5NO_4S$, which shows delocalized bonds in the six-membered ring. In the crystal, adjacent zwitterions are linked by N—H···O and O—H···O hydrogen bonds into a layer motif. The crystal studied was a racemic twin.

Related literature

A previous synthesis yielded hydronium 4-oxo-1,4-dihydropyridine-3-sulfonate dihydrate; see: Zhu *et al.* (2009).



Experimental

Crystal data

$C_5H_5NO_4S$
M_r = 175.16
Orthorhombic, $P2_12_12_1$
a = 6.7980 (2) Å
b = 8.7618 (3) Å
c = 10.6797 (3) Å
V = 636.11 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 293 K
0.28 × 0.23 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.880, T_{max} = 0.925
6216 measured reflections
1449 independent reflections
1403 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.068
S = 1.09
1449 reflections
121 parameters
5 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 785 Friedel pairs
Flack parameter: 0.31 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O2^{i}	0.83 (1)	1.76 (1)	2.581 (2)	166 (3)
N1—H1n···O3^{ii}	0.87 (1)	1.91 (1)	2.762 (2)	166 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (ii) $x+1, y, z$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (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, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2249).

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supporting information

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4-Hydroxypyridinium-3-sulfonate

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S1. Comment

A previous reaction of 4-hydroxpyridine and oleum gave the salt, hydronium 4-oxo-1,4-dihydropyridine-3-sulfonate dihydrate (Zhu *et al.*, 2009). Repeating this synthesis instead produced the zwitterionic title compound (Scheme I, Fig. 1). The bonds in the ring are delocalized bonds. Adjacent zwitterions are linked by N–H···O and O–H···O hydrogen bonds into a layer motif (Fig. 2).

S2. Experimental

4-Hydroxypyridine (10 mmol) was dissolved in 20% oleum (10 ml). The solution was heated to 393 K for 4 days. After it was cooled to room temperature, the excess oleum was decanted. Recrystallization of the solid from ethanol gave colorless crystals.

S3. Refinement

Carbon-bound H atoms were refind with a C–H 0.95±0.01 Å restraint. The amino and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints of O–H 0.84±0.01 Å and N–H 0.88±0.01 Å. All temperature factors were refined.

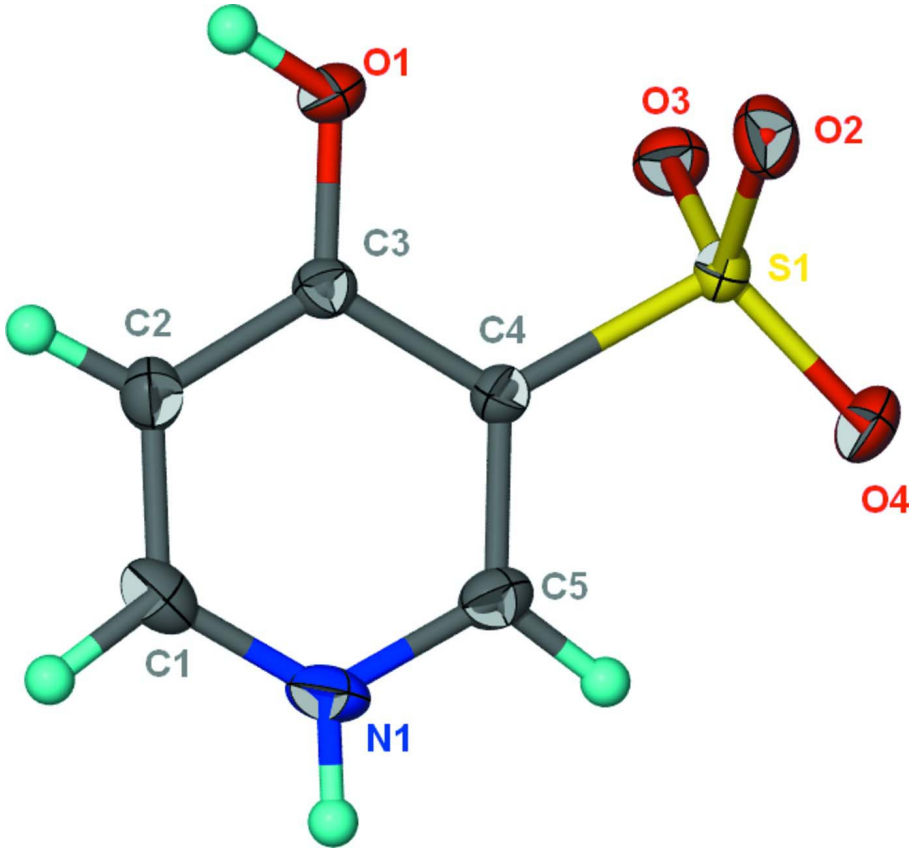

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_5H_5NO_4S$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

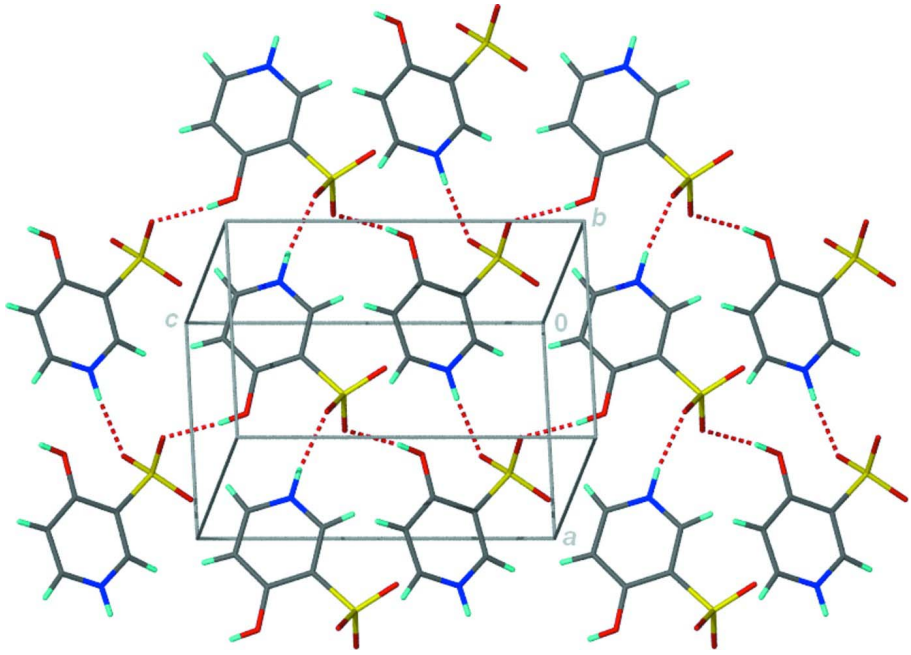

Figure 2

Hydrogen-bonded layer structure.

4-Hydroxypyridinium-3-sulfonate

Crystal data

$C_5H_5NO_4S$
$M_r = 175.16$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.7980$ (2) Å
$b = 8.7618$ (3) Å
$c = 10.6797$ (3) Å
$V = 636.11$ (3) Å^3
$Z = 4$
$F(000) = 360$
$D_x = 1.829$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6017 reflections
$\theta = 3.0$–$27.4°$
$\mu = 0.47$ mm^{-1}
$T = 293$ K
Prism, colorless
0.28 × 0.23 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.880$, $T_{max} = 0.925$
6216 measured reflections
1449 independent reflections
1403 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.0°$
$h = -8 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.068$
$S = 1.09$
1449 reflections
121 parameters
5 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
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.0899P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 785 Friedel pairs
Absolute structure parameter: 0.31 (8)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.84628 (5)	0.31587 (4)	0.15713 (3)	0.02340 (12)
O1	0.81623 (18)	0.44053 (16)	0.41523 (12)	0.0304 (3)
O2	0.7512 (2)	0.46465 (15)	0.13994 (12)	0.0348 (3)
O3	0.71610 (16)	0.20834 (15)	0.21966 (11)	0.0307 (3)
O4	0.9407 (2)	0.25936 (17)	0.04580 (11)	0.0344 (3)
N1	1.37517 (19)	0.33180 (18)	0.31720 (15)	0.0310 (3)
C1	1.3417 (3)	0.3866 (2)	0.43239 (17)	0.0317 (4)
C2	1.1563 (3)	0.42245 (19)	0.47075 (15)	0.0282 (3)
C3	0.9993 (2)	0.40609 (18)	0.38677 (14)	0.0224 (3)
C4	1.0389 (2)	0.34817 (17)	0.26608 (14)	0.0216 (3)
C5	1.2292 (2)	0.3108 (2)	0.23489 (15)	0.0274 (3)
H1O	0.794 (5)	0.485 (3)	0.4827 (16)	0.064 (9)*
H1N	1.4924 (18)	0.306 (3)	0.2930 (19)	0.036 (6)*
H1	1.456 (2)	0.394 (3)	0.4826 (19)	0.038 (6)*
H2	1.138 (3)	0.463 (2)	0.5519 (11)	0.021 (4)*
H5	1.267 (3)	0.263 (3)	0.1575 (14)	0.042 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02372 (18)	0.02877 (19)	0.01770 (17)	0.00307 (16)	−0.00047 (14)	−0.00191 (14)
O1	0.0236 (6)	0.0426 (7)	0.0249 (6)	0.0052 (5)	0.0026 (5)	−0.0082 (5)
O2	0.0429 (7)	0.0352 (7)	0.0262 (6)	0.0118 (5)	−0.0080 (6)	0.0003 (5)
O3	0.0259 (5)	0.0354 (6)	0.0308 (6)	−0.0021 (5)	0.0030 (5)	−0.0030 (5)
O4	0.0361 (6)	0.0460 (7)	0.0212 (5)	0.0012 (6)	0.0036 (5)	−0.0086 (5)
N1	0.0174 (6)	0.0363 (8)	0.0393 (8)	0.0029 (6)	0.0028 (5)	0.0047 (6)
C1	0.0258 (8)	0.0326 (8)	0.0368 (9)	−0.0014 (7)	−0.0071 (8)	0.0052 (7)
C2	0.0298 (8)	0.0321 (7)	0.0227 (7)	−0.0004 (8)	−0.0030 (7)	0.0001 (6)
C3	0.0211 (7)	0.0237 (7)	0.0224 (7)	0.0004 (6)	0.0014 (6)	0.0017 (5)
C4	0.0210 (7)	0.0249 (7)	0.0189 (6)	0.0016 (5)	0.0009 (5)	0.0011 (6)
C5	0.0253 (7)	0.0296 (7)	0.0273 (7)	0.0022 (7)	0.0063 (6)	0.0018 (7)

Geometric parameters (Å, º)

S1—O4	1.4390 (12)	N1—H1N	0.868 (10)
S1—O3	1.4549 (12)	C1—C2	1.362 (3)
S1—O2	1.4666 (13)	C1—H1	0.947 (10)
S1—C4	1.7747 (15)	C2—C3	1.402 (2)

O1—C3	1.316 (2)	C2—H2	0.943 (9)
O1—H1O	0.834 (10)	C3—C4	1.411 (2)
N1—C5	1.338 (2)	C4—C5	1.375 (2)
N1—C1	1.340 (2)	C5—H5	0.961 (10)
O4—S1—O3	115.32 (8)	C1—C2—C3	119.25 (16)
O4—S1—O2	113.52 (8)	C1—C2—H2	119.0 (13)
O3—S1—O2	111.40 (8)	C3—C2—H2	121.6 (13)
O4—S1—C4	105.51 (7)	O1—C3—C2	123.29 (15)
O3—S1—C4	104.55 (7)	O1—C3—C4	118.31 (14)
O2—S1—C4	105.41 (7)	C2—C3—C4	118.40 (15)
C3—O1—H1O	118 (2)	C5—C4—C3	119.11 (14)
C5—N1—C1	121.76 (14)	C5—C4—S1	119.83 (12)
C5—N1—H1N	116.7 (15)	C3—C4—S1	121.01 (11)
C1—N1—H1N	121.5 (15)	N1—C5—C4	120.37 (15)
N1—C1—C2	121.07 (16)	N1—C5—H5	115.2 (14)
N1—C1—H1	113.9 (15)	C4—C5—H5	124.4 (14)
C2—C1—H1	125.0 (15)		
C5—N1—C1—C2	0.4 (3)	O3—S1—C4—C5	118.79 (15)
N1—C1—C2—C3	−2.0 (3)	O2—S1—C4—C5	−123.66 (15)
C1—C2—C3—O1	−178.80 (16)	O4—S1—C4—C3	179.33 (13)
C1—C2—C3—C4	1.9 (2)	O3—S1—C4—C3	−58.62 (15)
O1—C3—C4—C5	−179.69 (15)	O2—S1—C4—C3	58.93 (15)
C2—C3—C4—C5	−0.4 (2)	C1—N1—C5—C4	1.2 (3)
O1—C3—C4—S1	−2.3 (2)	C3—C4—C5—N1	−1.2 (3)
C2—C3—C4—S1	177.04 (12)	S1—C4—C5—N1	−178.64 (12)
O4—S1—C4—C5	−3.26 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O2^{i}	0.83 (1)	1.76 (1)	2.581 (2)	166 (3)
N1—H1n···O3^{ii}	0.87 (1)	1.91 (1)	2.762 (2)	166 (2)

Symmetry codes: (i) −*x*+3/2, −*y*+1, *z*+1/2; (ii) *x*+1, *y*, *z*.