Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isonicotinium hydrogen sulfate

Li-Zhuang Chen

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: clz1977@sina.com

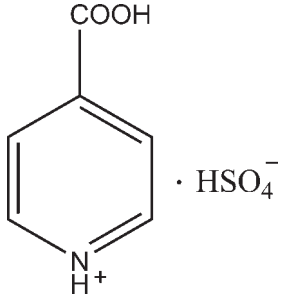
Received 10 August 2009; accepted 31 August 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.091; data-to-parameter ratio = 15.2.

The crystal structure of the title compound, $C_6H_6NO_2^+\cdot HSO_4^-$, is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For background to simple molecular–ionic crystals containing organic cations and acidic anions (1:1 molar ratio), see: Czupiński *et al.* (2002); Katrusiak & Szafrański (1999, 2006). For a related structure, see: Jebas *et al.* (2006).



Experimental

Crystal data

$C_6H_6NO_2^+\cdot HSO_4^-$
M_r = 221.18
Monoclinic, $P2_1/c$
a = 8.3816 (17) Å
b = 11.439 (2) Å
c = 9.4057 (19) Å
β = 109.12 (3)°
V = 852.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 293 K
0.25 × 0.22 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.90, T_{max} = 0.92
8697 measured reflections
1947 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
S = 1.14
1947 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O6^{i}	0.85	1.81	2.6425 (19)	166
O3—H3···O5^{ii}	0.94	1.75	2.6543 (18)	160
N1—H1A···O5	0.86	1.94	2.787 (2)	167

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

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: *SHELXL97*.

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: PV2195).

References

Czupiński, O., Bator, G., Ciunik, Z., Jakubas, R., Medycki, W. & Świergiel, J. (2002). *J. Phys. Condens. Matter*, **14**, 8497–8512.
Jebas, S. R., Balasubramanian, T. & Light, M. E. (2006). *Acta Cryst.* E**62**, o3481–o3482.
Katrusiak, A. & &Szafrański, M. (1999). *Phys. Rev. Lett.* **82**, 576–579.
Katrusiak, A. & Szafrański, M. (2006). *J. Am. Chem. Soc.* **128**, 15775–15785.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2349 [doi:10.1107/S1600536809034916]

Isonicotinium hydrogen sulfate

Li-Zhuang Chen

S1. Comment

Recently, much attention has been devoted to simple molecular–ionic crystals containing organic cations and acid radicals (1:1molar ratio) due to the tunability of their special structural features and their interesting physical properties (Czupiński *et al.*, 2002; Katrusiak & Szafrański, 1999; Katrusiak & Szafrański, 2006). The crystal structure of isonicotinium nitrate monohydrate has been reported. (Jebas *et al.*, 2006). In our laboratory, the title compound, (I), has been synthesized, its crystal structure is reported herein.

The asymmetric unit of the title compound consists of protoned isonicotinic acid $C_6H_6NO_2^+$ and HSO_4^-anions (Fig. 1). The isonicotinium cation is essentially planar. The crystal structure is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds. The H-bonds form a two-dimensional network as presented in Fig 2. viewed along the *a*-axis.

S2. Experimental

Isonicotinic acid (10 mmol) and 10% aqueous H_2SO_4 in a molar ratio of 1:1 were mixed and dissolved in water by heating to 353 K forming a clear solution. The reaction mixture was cooled slowly to room temperature, crystals of the title compound were formed, collected and washed with dilute aqueous H_2SO_4.

S3. Refinement

Hydrogen atoms bonded to N and O were located from a difference map and were included at those positions (O—H = 0.85/0.95 Å and N—H = 0.86 Å), while the remaining H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined using a riding model, with U_{iso}(H)=1.2U_{eq}(C,*N*) and 1.5U_{eq}(O).

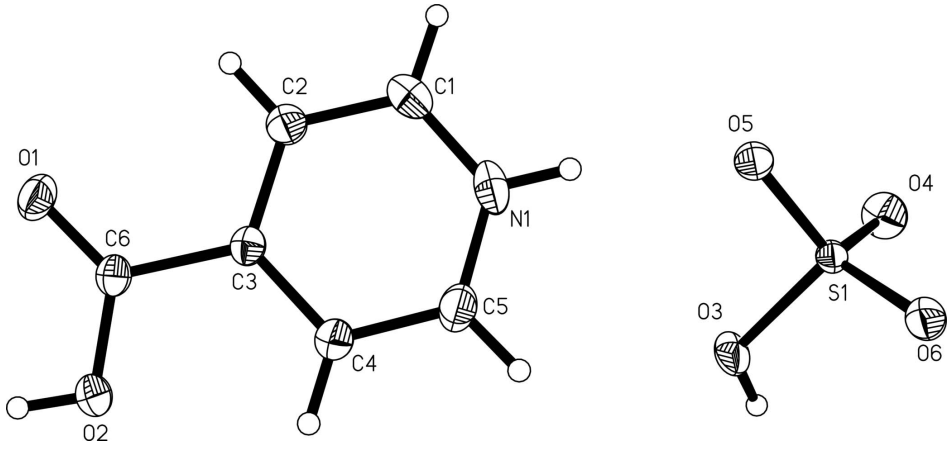


Figure 1

The asymmetric unit of the title compound with atom labels; the thermal elliposids have been drawn at 50% probability level.

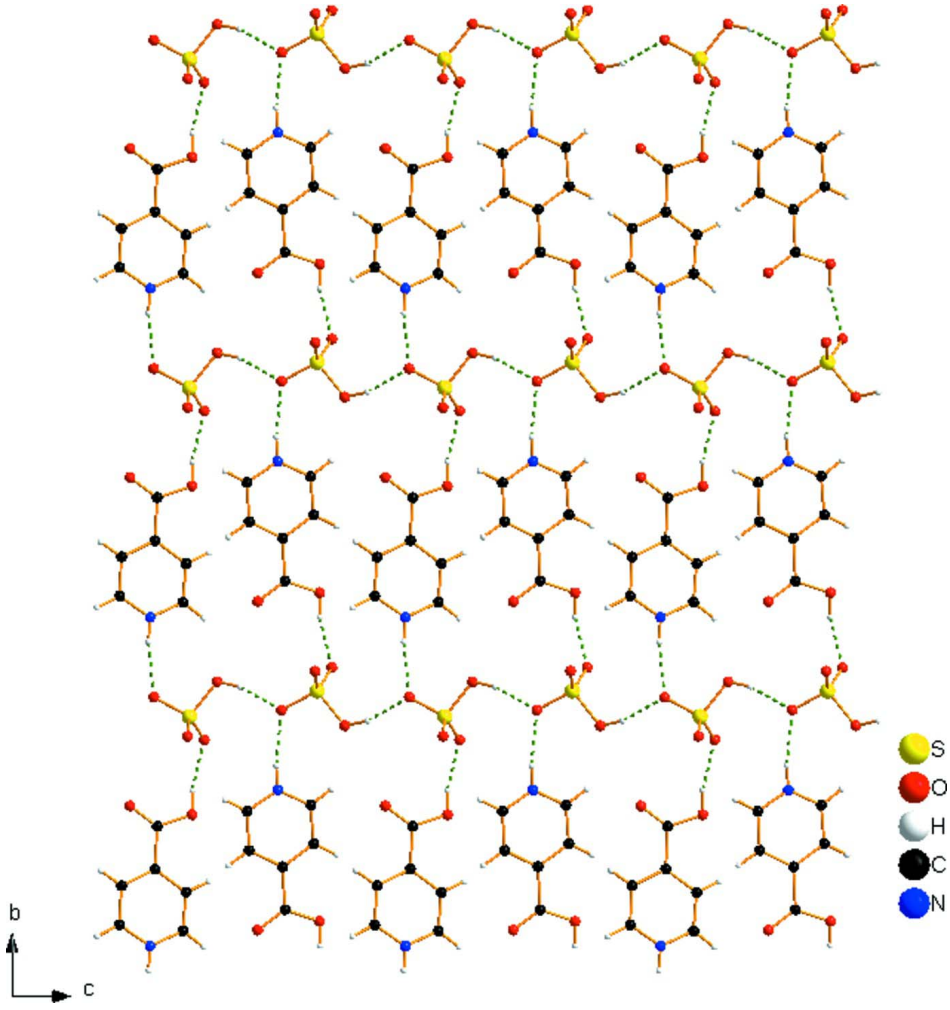


Figure 2

The H-bonded two-dimensional network of the title compound viewed down the *a* axis. Hydrogen bonds are drawn as dashed lines

Isonicotinium hydrogen sulfate

Crystal data

$C_6H_6NO_2^+ \cdot HSO_4^-$
$M_r = 221.18$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.3816\ (17)$ Å
$b = 11.439\ (2)$ Å
$c = 9.4057\ (19)$ Å
$\beta = 109.12\ (3)°$
$V = 852.0\ (3)$ Å^3
$Z = 4$

$F(000) = 456$
$D_x = 1.716$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1745 reflections
$\theta = 3.1–27.5°$
$\mu = 0.39$ mm^{-1}
$T = 293$ K
Block, colorless
0.25 × 0.22 × 0.2 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.90, T_{max} = 0.92
8697 measured reflections
1947 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −10→10
k = −14→14
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
S = 1.14
1947 reflections
128 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 0.395P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.164 (6)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.73722 (6)	0.21100 (4)	0.56236 (4)	0.02476 (17)
O2	0.86500 (19)	0.91218 (12)	0.57234 (16)	0.0404 (4)
H2B	0.8723	0.9863	0.5727	0.061*
O6	0.88648 (17)	0.14017 (12)	0.62022 (16)	0.0392 (4)
O1	0.71828 (19)	0.93897 (12)	0.32951 (16)	0.0405 (4)
O5	0.72948 (19)	0.26942 (12)	0.42296 (14)	0.0372 (4)
O4	0.58610 (18)	0.15100 (13)	0.55516 (17)	0.0427 (4)
O3	0.7598 (2)	0.31698 (11)	0.67114 (15)	0.0413 (4)
H3	0.7346	0.3019	0.7594	0.062*
C6	0.7789 (2)	0.87673 (16)	0.4359 (2)	0.0293 (4)
C3	0.7616 (2)	0.74617 (16)	0.42727 (19)	0.0270 (4)
C4	0.8443 (3)	0.67611 (17)	0.5487 (2)	0.0354 (4)
H4A	0.9152	0.7089	0.6374	0.042*
C2	0.6584 (2)	0.69616 (17)	0.2959 (2)	0.0346 (4)

H2A	0.6026	0.7427	0.2135	0.041*
N1	0.7200 (2)	0.51277 (15)	0.4078 (2)	0.0423 (4)
H1A	0.7067	0.4382	0.4019	0.051*
C5	0.8204 (3)	0.55833 (18)	0.5365 (2)	0.0418 (5)
H5A	0.8739	0.5099	0.6175	0.050*
C1	0.6393 (3)	0.57727 (19)	0.2881 (2)	0.0409 (5)
H1B	0.5707	0.5420	0.2002	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0360 (3)	0.0205 (2)	0.0191 (2)	−0.00064 (16)	0.01072 (17)	−0.00045 (14)
O2	0.0547 (9)	0.0241 (7)	0.0376 (8)	−0.0021 (6)	0.0087 (6)	−0.0019 (6)
O6	0.0381 (8)	0.0309 (7)	0.0407 (8)	0.0034 (6)	0.0023 (6)	−0.0038 (6)
O1	0.0506 (9)	0.0312 (7)	0.0398 (8)	0.0061 (6)	0.0148 (7)	0.0096 (6)
O5	0.0644 (10)	0.0292 (7)	0.0217 (7)	−0.0011 (6)	0.0194 (6)	−0.0001 (5)
O4	0.0385 (8)	0.0453 (9)	0.0477 (9)	−0.0067 (6)	0.0185 (6)	0.0008 (7)
O3	0.0794 (11)	0.0246 (7)	0.0280 (7)	−0.0030 (7)	0.0285 (7)	−0.0051 (5)
C6	0.0293 (9)	0.0258 (9)	0.0345 (10)	0.0018 (7)	0.0128 (7)	0.0022 (7)
C3	0.0284 (9)	0.0258 (9)	0.0294 (9)	0.0011 (7)	0.0130 (7)	0.0014 (7)
C4	0.0413 (11)	0.0296 (9)	0.0332 (10)	0.0013 (8)	0.0094 (8)	0.0017 (8)
C2	0.0341 (10)	0.0337 (10)	0.0343 (10)	0.0003 (8)	0.0090 (8)	0.0002 (8)
N1	0.0540 (11)	0.0234 (8)	0.0569 (12)	−0.0060 (7)	0.0282 (9)	−0.0041 (7)
C5	0.0550 (13)	0.0302 (10)	0.0412 (12)	0.0032 (9)	0.0171 (10)	0.0065 (9)
C1	0.0397 (11)	0.0389 (11)	0.0437 (12)	−0.0079 (9)	0.0133 (9)	−0.0110 (9)

Geometric parameters (Å, º)

S1—O4	1.4226 (15)	C3—C4	1.382 (3)
S1—O6	1.4393 (14)	C4—C5	1.361 (3)
S1—O5	1.4540 (13)	C4—H4A	0.9300
S1—O3	1.5574 (13)	C2—C1	1.369 (3)
O2—C6	1.314 (2)	C2—H2A	0.9300
O2—H2B	0.8500	N1—C1	1.331 (3)
O1—C6	1.197 (2)	N1—C5	1.334 (3)
O3—H3	0.9372	N1—H1A	0.8600
C6—C3	1.500 (3)	C5—H5A	0.9300
C3—C2	1.379 (3)	C1—H1B	0.9300
O4—S1—O6	113.36 (9)	C5—C4—H4A	120.5
O4—S1—O5	113.76 (9)	C3—C4—H4A	120.5
O6—S1—O5	112.10 (9)	C1—C2—C3	119.23 (19)
O4—S1—O3	108.75 (9)	C1—C2—H2A	120.4
O6—S1—O3	106.64 (9)	C3—C2—H2A	120.4
O5—S1—O3	101.22 (8)	C1—N1—C5	123.11 (18)
C6—O2—H2B	109.1	C1—N1—H1A	118.4
S1—O3—H3	115.1	C5—N1—H1A	118.4
O1—C6—O2	125.44 (18)	N1—C5—C4	119.67 (19)

O1—C6—C3	122.68 (17)	N1—C5—H5A	120.2
O2—C6—C3	111.87 (15)	C4—C5—H5A	120.2
C2—C3—C4	119.86 (18)	N1—C1—C2	119.18 (19)
C2—C3—C6	118.85 (16)	N1—C1—H1B	120.4
C4—C3—C6	121.28 (16)	C2—C1—H1B	120.4
C5—C4—C3	118.95 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*B*···O6^{i}	0.85	1.81	2.6425 (19)	166
O3—H3···O5^{ii}	0.94	1.75	2.6543 (18)	160
N1—H1*A*···O5	0.86	1.94	2.787 (2)	167

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