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Bis(2-aminopyridinium) sulfate

Samuel Robinson Jebas[a] Thailampillai Balasubramanian,[a]* Rene Peschar[b] and Jan Fraanje[b]

[a]Department of Physics, National Institute of Technology, Tiruchirappalli 620 015, India, and [b]Laboratory of Crystallography, Van't Hoff Institute for Molecular Sciences, University of Amsterdam, Valckenierstrasse 65,1018XE, Amsterdam, The Netherlands

Correspondence e-mail: bala@nitt.edu

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.086
Data-to-parameter ratio = 8.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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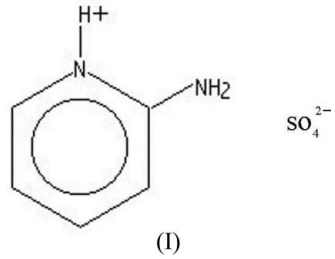
In the structure of the title compound, $2C_5H_7N_2^+\cdot SO_4^{2-}$, the S atom of the sulfate anion lies on a twofold axis. The structure is stabilized by an extensive network of N—H···O hydrogen bonds.

Comment

2-Aminopyridine is used in the manufacture of pharmaceuticals, especially antihistaminic drugs (Windholz, 1976). As a part of our investigations of inorganic salts of 2-aminopyridine, we report here the crystal structure of bis(2-aminopyridinium) sulfate (I) (Fig. 1 and Table 1).



(I)

The asymmetric unit of (I) contains two independent fragments, a 2-aminopyridinium cation and one half of a sulfate anion; the S atom lies on a twofold axis. Protonation of atom N1 of 2-aminopyridine results in a widening of the C2—N1—C6 angle to 122.7 (2)°. This compares to 117.7 (1)° in neutral 2-aminopyridine (Chao *et al.*, 1975). The bond lengths and angles in (I) are comparable to those in other 2-aminopyridinium complexes (Bis *et al.*, 2005; Smith *et al.*, 2000). The pyridinium ring is essentially planar, with a maximum deviation from the mean plane of 0.009 (3) Å for atom C3.

The crystal packing is stabilized by a series of N—H···O hydrogen bonds (Table 2 and Fig. 2) in a hydrogen-bonding pattern similar to those found in 2-aminopyridinium carboxylate complexes (Bis *et al.*, 2005) and cytosine carboxylates (Ohki *et al.*, 1975; Tamura *et al.*, 1972).

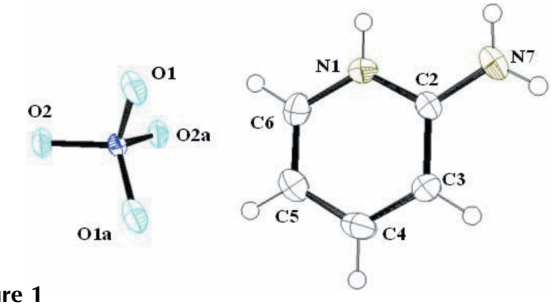


Figure 1
View of (I), shown with 50% probability displacement ellipsoids. [Symmetry code: (a) $\frac{1}{2}-x, \frac{1}{2}-y, z$.]

Experimental

Solutions of 2-aminopyridine and sulfuric acid were mixed in 1:1 molar ratio in water and heated at 363 K for 5 h. Colourless crystals of (I) were obtained by slow evaporation over a period of one week.

Crystal data

$2C_5H_7N_2^+\cdot SO_4^{2-}$
M_r = 286.31
Orthorhombic, *Fdd*2
a = 12.873 (7) Å
b = 16.835 (8) Å
c = 12.035 (8) Å
V = 2608 (3) Å^3
Z = 8
D_x = 1.458 Mg m^{-3}
Cu *Kα* radiation
μ = 2.39 mm^{-1}
T = 293 (2) K
Block, colourless
0.5 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.513, T_{max} = 0.550
702 measured reflections
702 independent reflections
698 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 74.6°
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.08
702 reflections
88 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.5871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0017 (2)

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.4785 (16)	N1—C2	1.345 (3)
S1—O1	1.4812 (18)	N1—C6	1.366 (4)
C2—N1—C6	122.7 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.86	2.701 (3)	165
N7—H7*A*···O2^{i}	0.86	2.15	2.923 (3)	150
N7—H7*B*···O2^{iii}	0.86	2.14	2.985 (3)	168

Symmetry codes: (i) $-x+\frac{1}{4}, y-\frac{1}{4}, z-\frac{1}{4}$; (ii) $x, y-\frac{1}{2}, z-\frac{1}{2}$; (iii) $x+\frac{1}{4}, -y+\frac{1}{4}, z-\frac{3}{4}$.

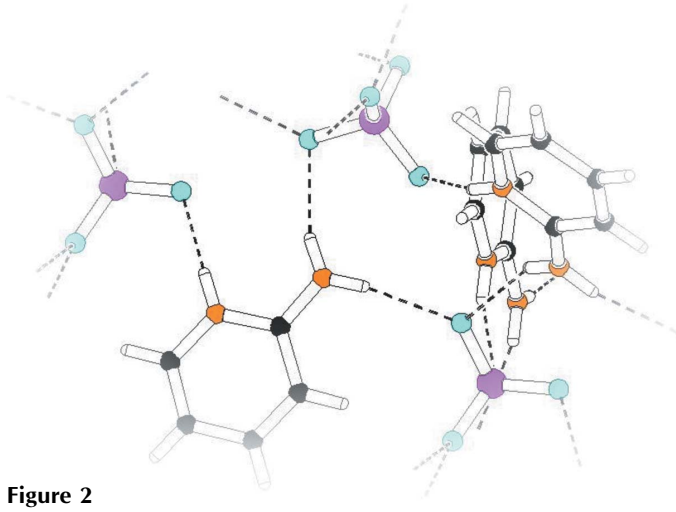


Figure 2
The hydrogen-bonding pattern for (I). Hydrogen bonds are drawn as dashed lines.

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. H atoms were placed in calculated positions, with C—H = 0.93 Å and N—H = 0.86 Å, nd refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,*N*).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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