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Hydrogen-bonding patterns of 2-aminopyridinium *p*-nitrobenzoate

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In the title compound, $C_5H_7N_2^+\cdot C_7H_4NO_4^-$, the asymmetric unit consists of an aminopyridinium cation and a *p*-nitrobenzoate anion, with proton transfer occurring to the aromatic N atom of the 2-aminopyridine molecule. In the crystal structure, the two ions are interconnected by N—H···O hydrogen bonds.

Key indicators

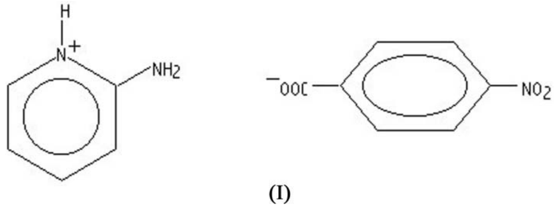
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.129
Data-to-parameter ratio = 8.5

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

Comment

para-Aminopyridine is a commonly used drug for the treatment of neurological ailments, such as multiple sclerosis, with tests showing that *p*-aminopyridine improves motor functions in multiple sclerosis patients (Schwid *et al.*, 1997). In episodic ataxia type 2, it functions as a potassium channel blocker (Strupp *et al.*, 2004).

As an extension of our systematic study of hydrogen-bonding patterns of 2-aminopyridine with aromatic carboxylic acids, the title compound, (I) has been synthesized and the crystal structure determined.



(I)

2-Aminopyridine and *p*-nitrobenzoic acid form a salt, with the proton from the carboxyl group transferred to atom N1 of 2-aminopyridine, thereby leading to widening of the corresponding internal angle (C1—N1—C5) of the pyridine ring to 121.9 (3) Å, compared with 117.7 (1) Å in the unprotonated 2-aminopyridine (Chao *et al.*, 1975). Similar protonation is observed in various 2-aminopyridine acid complexes (Joanna & Zaworotko, 2005; Smith *et al.*, 2000). The distances and angles in complex (I) are comparable to those in various 2-aminopyridine acid complexes (Joanna & Zaworotko, 2005).

The crystal structure is maintained through a series of N—H···O hydrogen bonds with N···O distances in the range 2.686 (4)–2.814 (4) Å. Protonated 2-aminopyridine forms a fork-like hydrogen bonding pattern with the carboxylate group. This fork-like interaction is observed in cytosine carboxylate interactions (Ohki *et al.*, 1975; Tamura *et al.*, 1972). The nature of the interaction and the mode of hydrogen bonding are quite similar to those found in 2-aminopyridinium carboxylate complexes (Joanna & Zaworotko, 2005).

The rings are essentially planar, with a maximum deviation of the ring atoms from the least-squares plane of 0.014 (1) Å for C1 in the 2-aminopyridinium cation and 0.007 (2) Å for C9 in the *p*-nitrobenzoate anion. The bond lengths and angles of the *p*-nitrobenzoate anion are found to be normal (Andrau & White, 2003).

Experimental

Solutions of 2-aminopyridine and *p*-nitrobenzoic acid were mixed in 1:1 molar ratio in methanol and warmed in a water bath for 2 h. Colourless crystals were obtained after a week *via* slow evaporation.

Crystal data

$C_5H_7N_2^+ \cdot C_7H_4NO_4^-$
$M_r = 261.24$
Monoclinic, $P2_1$
$a = 7.9403\ (12)$ Å
$b = 6.7719\ (6)$ Å
$c = 11.1120\ (15)$ Å
$\beta = 99.272\ (6)°$
$V = 589.70\ (13)$ Å^3
$Z = 2$
$D_x = 1.471$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
$0.3 \times 0.13 \times 0.06$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
7208 measured reflections
1463 independent reflections
1035 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.071$
$\theta_{\rm max} = 27.6°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.129$
$S = 1.05$
1463 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.0892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.036$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.041 (11)

Table 1
Selected geometric parameters (Å, °).

O1—C13	1.264 (4)	C6—N3	1.470 (5)
O4—N3	1.230 (4)	C1—N2	1.324 (4)
O3—N3	1.236 (4)	C1—N1	1.353 (4)
O2—C13	1.253 (4)	C5—N1	1.360 (4)
C7—C6—N3	119.1 (3)	O4—N3—C6	118.8 (3)
O1—C13—O2	125.0 (3)	O3—N3—C6	118.4 (3)
O1—C13—C9	116.9 (3)	N2—C1—N1	118.3 (3)
O2—C13—C9	118.1 (3)	N2—C1—C2	123.6 (3)
O4—N3—O3	122.8 (3)	C1—N1—C5	121.9 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1$^{\rm i}$	0.86	1.83	2.686 (4)	176
N2—H2A···O2$^{\rm i}$	0.86	1.95	2.809 (4)	176
N2—H2B···O2$^{\rm ii}$	0.86	1.97	2.814 (4)	168

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

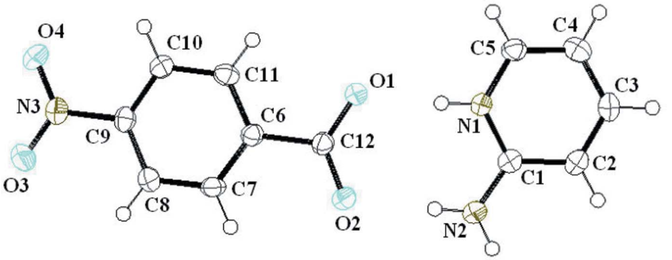


Figure 1
View of (I), with 50% probability displacement ellipsoids.

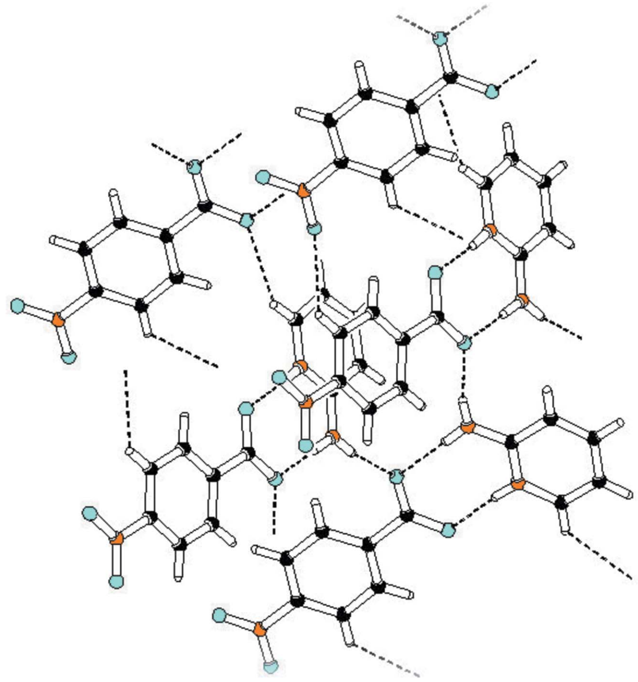


Figure 2
Fork-like hydrogen-bonding (dashed lines) pattern of (I).

H atoms were placed in calculated positions, with C—H = 0.93 Å and N—H= 0.86 Å, and refined using a riding model, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N). Friedel pairs were merged. The number of Friedel pairs measured was 1133. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; 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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